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Report _____

IMPROVED PLAQUE MATERIALS FOR AEROSPACE NICKEL-CADMIUM CELLS

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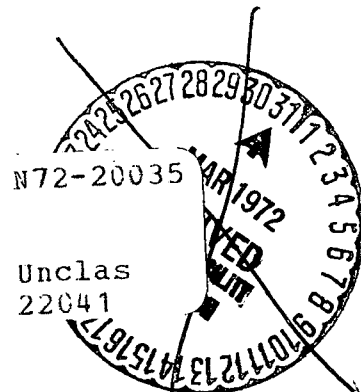
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16. Abstract <p>Improved cadmium electrode substrates with precisely controlled microstructures for possible use in aerospace nickel-cadmium cells were prepared. The preparative technique was a powder metallurgical process in which a fugitive pore-former and a nickel powder were blended, then isostatically compacted, and subsequently sintered. Cadmium electrodes prepared from such substrates were cycle tested using an accelerated tortuous test regime. It was discovered that plaques of 60% or 80% porosity prepared with a 25μ pore-former were better than state-of-the-art electrodes in terms of efficiency and/or mechanical strength. The 60% structures were particularly outstanding in this respect in that they had efficiencies only 5-10 percentage points lower than state-of-the-art electrodes and vastly superior mechanical properties. This added strength was observed to eliminate cracking and physical degradation of the electrodes during processing and cycling. The cadmium electrodes prepared from the 80% porous substrates proved to be the best electrodes made during the course of the work from the point of view of highest efficiency. These were also somewhat stronger than conventional plaques and had better uniformity.</p> <p>Three-point bend tests were used to measure mechanical properties of the plaques produced and also as a general characterization tool. In addition, the BET surface areas of selected specimens was determined in order to help along toward this end.</p> <p>The SEM was used for judging microscopic uniformity and quantitatively determining the induced pore size and various other fine structures in the substrates.</p> <p>The technique of X-ray radiography was used to follow the bulk uniformity of the above-mentioned substrates at various stages of their processing into cadmium electrodes and cycle testing.</p>			
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PREFACE

This report summarizes exploratory work conducted at the Gould Energy Technology Laboratories on a novel process for the preparation of improved cadmium electrode plaques with precisely controlled pore size and porosity for use in aerospace nickel-cadmium cells. The requirements for these aerospace components include far better uniformity, reproducibility, and life than is currently available with conventional materials. The key features of the work was preparation of plaques via a powder metallurgical technique which consisted of isostatically compacting a blend of a nickel powder with a fugitive pore-former and subsequently sintering the "green" compact. Cadmium electrodes were prepared from these materials and cycle tested in negative limited cells using an accelerated, tortuous test regime. Results were obtained which showed the new structures yielded electrodes that were superior to the conventional structures in mechanical properties and performance. BET surface areas, SEM photomicrographs, X-ray radiographs, and mechanical strengths using a three-point bend test were used to help in the thorough characterization of the electrodes and the components prepared. The improved properties and performance of cadmium electrode prepared from isostatically compacted plaques suggests further studies for optimizing their structure.

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I. INTRODUCTION

There is currently a need for very long lived highly reliable secondary batteries for use in space vehicles. Nickel-cadmium batteries of the sealed type are uniquely suited for such application mainly because of their long lives if employed under the proper set of operating conditions. Under tortuous conditions the utility of nickel-cadmium batteries becomes severely limited because cells can fail due to any number of factors, a very common one being cell rupture due to hydrogen generation on overcharge. This is a result of aging effects on both electrodes and especially capacity degradation on the cadmium electrode under very tortuous operating conditions.

Such failures have been blamed on lack of uniformity and reproducibility of the battery components. This is reasonable, for if components are not uniform and reproducible, it is not possible to effectively over-design for the short-comings of the system.

Past efforts to produce uniform materials for aerospace cells have been concerned with bulk or macroscopic uniformity. That is, special attention was given to produce, for example, plaques and electrodes of thickness, loading, porosity, etc. within tighter limits. In spite of this, electrodes that were apparently uniform on a bulk basis have shown widely differing aging characteristics indicating that perhaps not all key variables have been considered.

Rather than concentrating on macroscopic uniformity, in the work discussed herein the uniformity on the microscopic scale was controlled, and its role on the life and aging characteristics were evaluated by testing highly porous plaques of very closely controlled porosity, pore-size, and pore distribution. Since this exploratory program was for the purpose of studying microscopic uniformity, minimum effort was made to produce structures of very high degree of bulk uniformity other than to show that uniform structure could be made by the technique used and assure that results were not confounded by these parameters. Nevertheless, materials with very good but not optimum uniformity were produced. It was felt that the ultimate in uniformity should be pursued after the merit of the process was established.

To this end a powder metallurgical technique was chosen to prepare uniform plaques because these are the most versatile and direct processes for the fabrication of porous materials suitable for practical electrode components. A pore-former type process was selected as holding forth the most promise. With the use of a pore-former, one can precisely control both induced and interstitial porosity to tailor-make a structure to meet a specific application.

This paper distinguishes between two types of porosity. Interstitial porosity is the naturally occurring void volume between neighboring particles in a more or less close packed aggregate. Induced porosity, on the other hand, is formed by the addition of a fugitive pore-former to the unconsolidated powder. An inter-connected void volume is subsequently formed by the removal of the added powder.

In the packing of powder particles, particularly in the case of fine or irregularly shaped powders, a form of gross packing imperfection called bridging will occur. Bridging is the formation of an arch or bridge of powder particles producing larger voids within the aggregate and, it is, in part, the cause of the low packing densities of uncompacted high surface area powders. The void volumes, or pore, formed by a bridge may have a vastly greater number of particles surrounding it than in the case of an interstitial pore, and the pore diameter is not directly related to particle diameter.

Induced porosity is caused by the bridging of powder particles caused by the introduction of the pore-forming agent to the powder. The powder is packed about the particles of pore-former, which is subsequently removed by decomposition, solution, or vaporization. As a result, the character of induced porosity is a function of the particle size distribution, volume percent, and surface area of pore-former added to the powder. The pore-former diameter may be orders of magnitude larger than the metal particle diameter and the porosity can exceed 90 %, depending upon the size and strength of the powder particle bridges. It should be appreciated, however, the interstitial porosity between the particles in a bridge or wall surrounding an induced pore can be maintained, and the porosity of an induced pore retains some interstitial character. For example, the large induced pores may be interconnected by means of the fine interstitial pores in the pore wall or bridge, and the total surface area of the compact may include the interstitial pore area. The large pores may also be mutually interconnected for greater permeability.

To prepare an induced-pore electrode plaque, a nickel powder previously screened to yield an average particle diameter of the desired size, and a powdered pore-former of the proper particle size to form pores of the necessary diameter was blended with the nickel powder. The blended powder mixture was then compacted to cold-sinter the nickel particles. The pore-former was removed from the compact leaving a structure that was subsequently sintered at the desired time and temperatures. The following features of the resulting sintered plaque which are illustrated in Figure 1 were especially significant:

- The pore diameter can be any size, some 100 times larger than the metal particle size if required
- The pore size is uniform, reflecting the size distribution of the pore-former
- The pore wall contains any level of interstitial porosity so that all the particle surface is available to the large pores

Pressure can be applied to the powder by means of a number of techniques. For example, uniaxially in a die, isostatically in a flexible mold in a pressurized fluid, or in a rolling mill. Because a powder does not behave in an ideal hydrodynamic manner under pressure, a consequence of interparticles and die wall friction, die pressing usually results in variations in microstructure throughout the compact. These variations are particularly severe in extremely thin compacts. Uniform microstructures can best be achieved by isostatic pressing.

In ordinary die pressing, the friction between powder particles and between powder particles and die walls prevents uniform compaction. The powder becomes highly compacted in areas adjacent to the die and punch surfaces and is somewhat less dense toward the interior of the compacted part. Further, in die compaction the microscopic high points after the powder is struck-off reflect themselves as areas of high densities.

The primary objective of the program was the exploration of isostatic compaction techniques to product thin, polyporous nickel plaque materials for cadmium electrodes of nickel-cadmium batteries for aerospace applications. However, in the early phase of the program plaques were prepared using die compaction techniques*. Thin plaques produced by the die compaction method were not as uniform as commercial material and further work along this line of approach was discontinued in order to devote the entire effort on isostatic compaction.

The isostatic compaction process used was the so-called wet-bag process. The process is illustrated in outline form in Figure 2. In the work reported herein, parts were compacted, via pressurized liquid against rubber diaphragms, from two directions. A detailed description of the process is given in the Experimental Section.

*Subcontract to North American Rockwell Corp., Rocketdyne Division

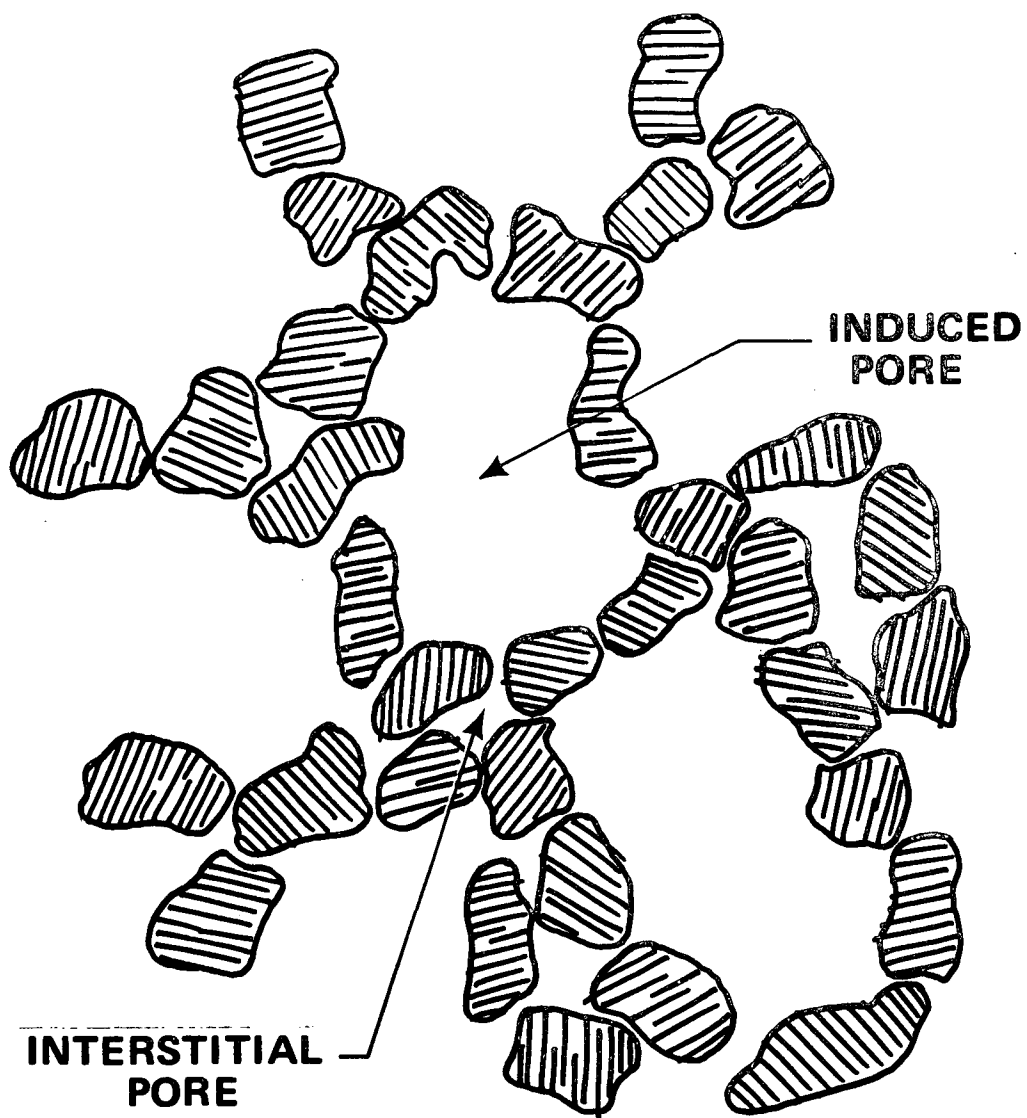


FIGURE 1. INDUCED POROSITY NICKEL SUBSTRATE

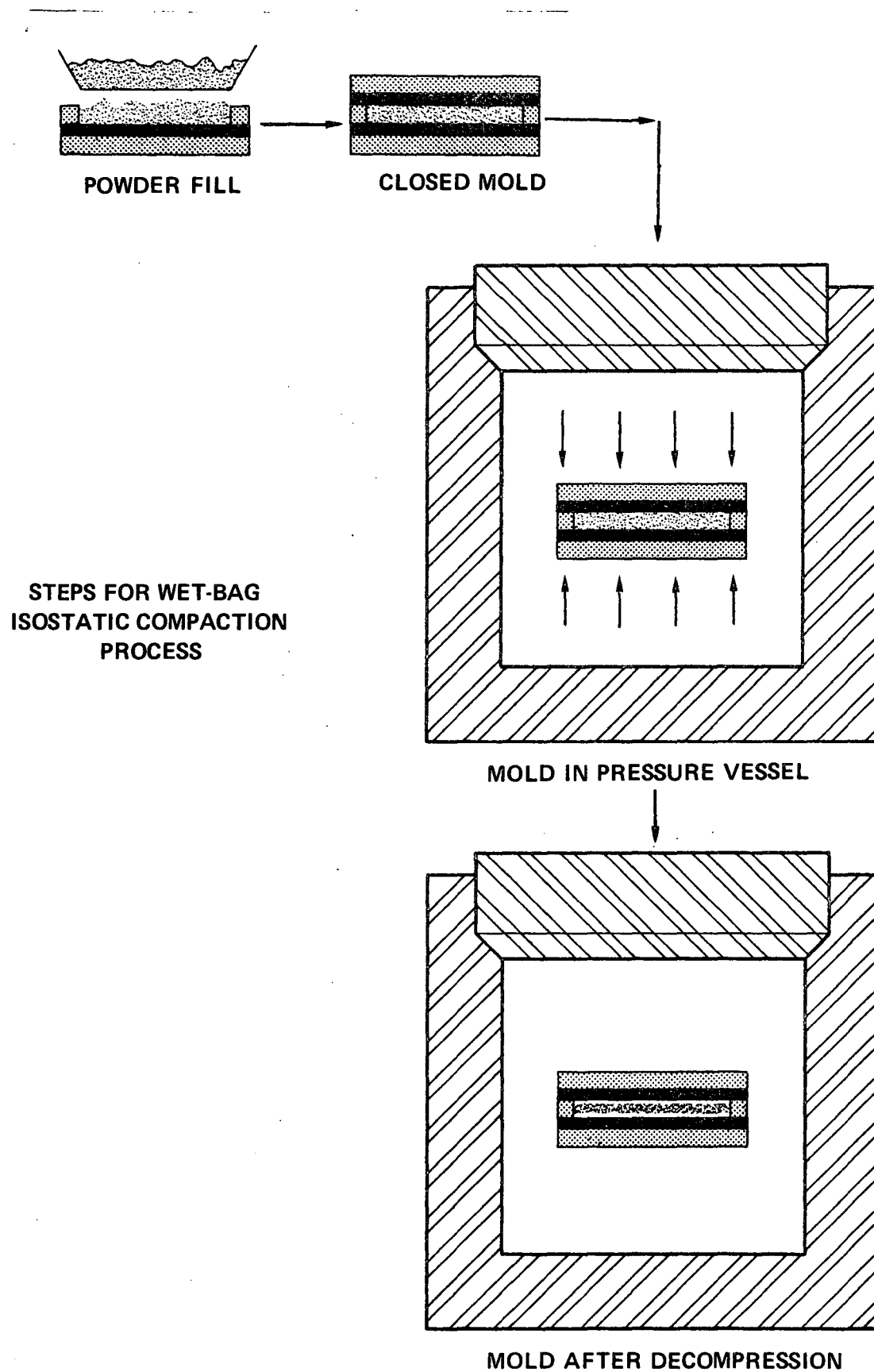


FIGURE 2. STEPS FOR WET-BAG ISOSTATIC COMPACTION PROCESS

II. EXPERIMENTAL

The experimental work involved pore-former preparation, nickel powder preparation, blending of the two powders, compaction of the powder blend, removal of the pore-former, sintering of the "green" plaque, and electrode fabrication and testing. The following is a brief outline of these procedures.

A. PLAQUE PREPARATION

Figure 3 illustrates the process flow of the various operations required in the fabrication of the plaques. Each operation is described in some detail in the paragraphs that follow.

1. **Pore-Former:** Of the various materials considered in this program as candidates for use as pore-formers (sodium chloride, urea, thiosemicarbazide, camphor), $(\text{NH}_4)_2\text{CO}_3$ was found to be the most convenient material to use for this purpose. It was prepared as follows:

- Fisher purified grade powder was placed in a pan in a dry room (R.H. 3-5%) overnight to allow loss of any excess moisture.
- The powder was then placed in a porcelain ball mill and ground for 15 to 20 minutes utilizing 13/16 in. diameter Burundum grinding media.
- After grinding, the powder was placed in a stack of standard sieves and placed on a sieve shaker for 15 minutes. The -37μ fraction was separated and placed aside, and the remaining fractions were labeled and saved for future use and/or grinding. At this point in the processing, samples of each lot of $(\text{NH}_4)_2\text{CO}_3$ used were taken for density determination. Results for the four lots of powder used, as determined on an air pycnometer were: 1.659 g/cc, 1.614 g/cc, 1.653 g/cc, and 1.575 g/cc. This gives an average value for all lots of 1.625 ± 0.062 g/cc. The narrow range of densities shows the materials were adequate for their intended purpose.

When a sufficient fraction of -37μ powder was obtained, it was placed in a three-inch micro-mesh sieve stack (44μ , 30μ , 20μ , 10μ) and placed on a shaker for 15 minutes. The various fractions were then stored in separate labeled jars until needed. All operations on the pore-formers (grinding, sieving, and storage) were performed in a dry room (R.H. 3-5%).

2. **Nickel Powder:** Inco 287 powder was used in this work except where otherwise stated. It was prepared by placing a sample in a vacuum oven for one hour at 210°C . After removal from the oven, the powder was placed in the dry room to cool. When cool, the powder was placed in the standard sieves and processed on the shaker for 15 minutes. The -37μ (400 mesh) fraction was stored in the dry room and the remaining fractions discarded.
3. **Powder Blending:** To prepare a blend for an 80 % porous structure, for example, the nickel and $(\text{NH}_4)_2\text{CO}_3$ were weighed out on a Mettler P162 balance to obtain Ni: $(\text{NH}_4)_2\text{CO}_3$ ratio of 1.5:1 by weight, the ratio required to yield a 80 % plaque. The normal working weight was 200 grams. This was used to aid in handling and to avoid changes in composition due to storage. The powders were then mixed and placed in a standard sieve (the sieve used was one size larger than the pore-former fraction used, except for the sizes smaller than 37μ for which the 400-mesh sieve was

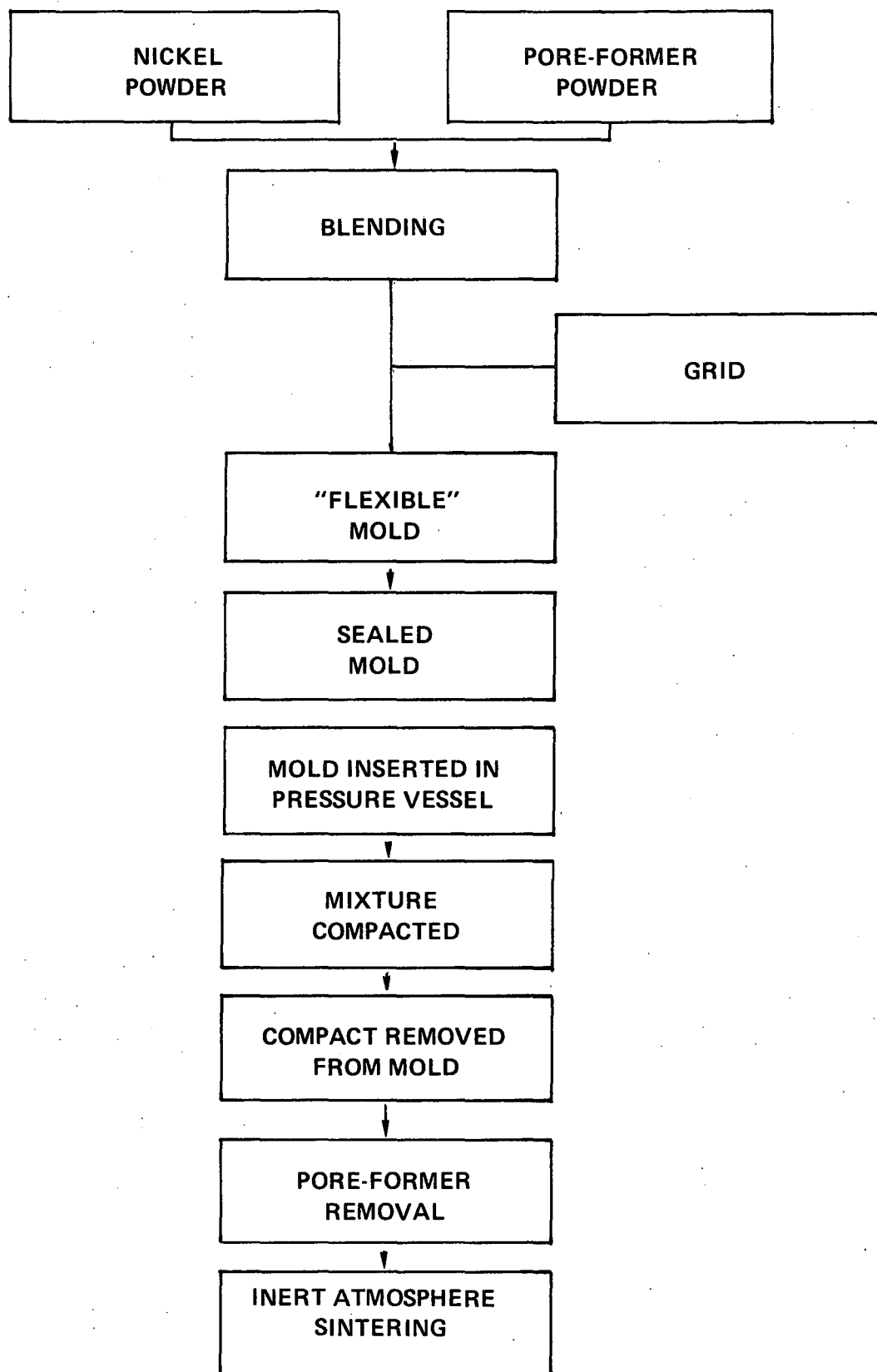


FIGURE 3. PROCESS FLOW CHART, PLAQUE FABRICATION

employed) and brushed through with a bristle paintbrush. This procedure was followed to minimize lumping of the pore-former. Following the brushing process, the blend was placed in a Patterson-Kelley twin-shell dry blender and allowed to mix for 30 minutes. After mixing, the material was again brushed through a sieve and then stored in the dry room. All the above operations were carried out in the dry room. The blend for a 60 % porous structure was prepared in a similar manner except that the Ni to $(\text{NH}_4)_2\text{CO}_3$ ratio was 9:1, the value required to prepare a plaque of 60 % porosity.

4. **Powder Compaction:** The blend was then loaded into the cavity of a flexible mold (Figure 4) which already contained an annealed 20-mesh, 7-mil wire, woven screen, by brushing through an appropriate size sieve (for the 37μ size pore-former, a 45μ sieve was used). The essential feature of this mold was the two flexible ends which permitted compaction, via the rubber parts, from both sides of the specimen. This mold allowed the preparation of a part three inches in diameter. All excess material was struck off, and the mold closed and inserted into an Autoclave Engineers four-inch isostatic press and compacted at a pressure of 7000 psi for one minute.
5. **Pore-Former Removal:** After compaction, the "green" sintered specimen was removed from the mold and the $(\text{NH}_4)_2\text{CO}_3$ pore-former was removed by placing the sample in a vacuum oven and decomposing under vacuum at 210°C for 20 minutes.
6. **Green Plaque Sintering:** The "green" plaques were then removed from the decomposition oven and immediately placed into a vacuum furnace where they were sintered in a TM Vacuum Products oven at a pressure of less than 50μ at 1600°F for 10 minutes. Helium was used as a back fill and cooling media.
7. **Plaque Inspection:** After sintering, all plaques were examined visually for irregularities, trimmed, and weighed and measured to determine their apparent densities.

Selected specimens were analyzed in terms of geometry (thickness, porosity, etc.), pore-size, (from photomicrographs), mechanical strength, and uniformity as judged from X-ray radiographs.

B. PLAQUE CHARACTERIZATION

1. **Plaque Geometry:** Plaque dimensions in this study were 2.35×1.62 in. and approximately 30 mils thick. The thickness variation within one sample was determined by multiple readings with a micrometer with 0.8 inch diameter anvil and the average value used to determine the variation from sample to sample. Also recorded were screen weights and powder weights for each sample prepared.
2. **Mechanical Testing:** Mechanical strengths were measured for selected specimens on an Instron Tensile Tester by a three-point bend method.
3. **Photomicrographs:** Scanning electron photomicrographs of selected specimens were taken and measurements made on them to determine induced pore size and ratio of induced to interstitial porosity. Examination of photomicrographs was also used as a criteria for microscopic uniformity, along with such items as $\text{Cd}(\text{OH})_2$ crystal size and distribution, in virgin and cycled electrodes.

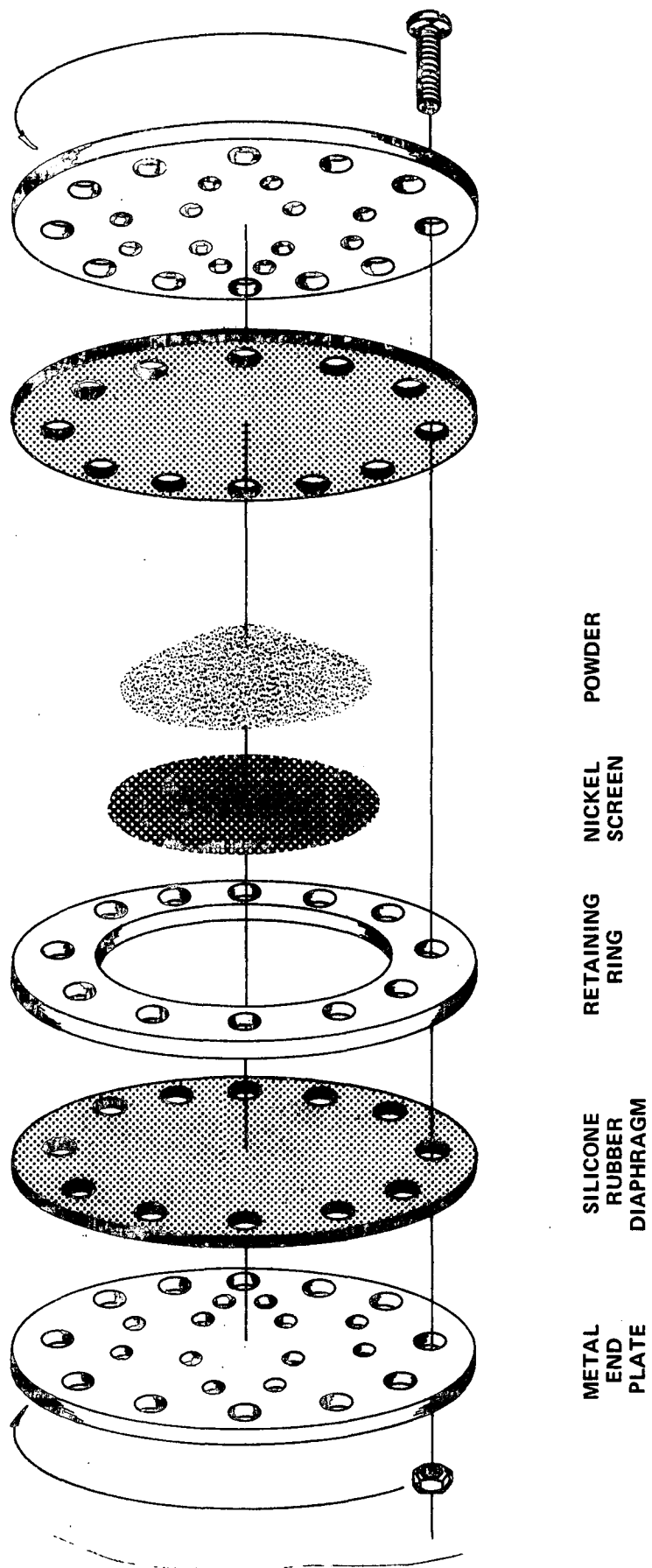


FIGURE 4. EXPLODED VIEW OF FLEXIBLE MOLD USED TO
ISOSTATICALLY COMPACT POWDERS

4. **X-Ray Radiographs:** Qualitative examination of x-ray radiographs was employed as an important technique for judging the macroscopic uniformity of all specimens at various stages of preparation and testing.
5. **B.E.T. Surface Area:** Surface areas of selected sintered plaques were determined with a Numinco-Orr Surface Area Pore-Volume Analyzer, Model 2100, using Krypton as the adsorbate. Surface areas were utilized for comparison with other properties.

C. CADMIUM ELECTRODE PROCESSING

The structures prepared as described above were impregnated by a proprietary process¹ in a manner that minimizes plaque damage. All electrodes in this study were loaded to 40.183 ± 0.883 g CdO per cubic inch of plaque (0.507 ± 0.031 Ah/in.² Cd(OH)₂ loading). It was felt that a constant loading per cubic inch of free space would yield electrodes far too low in energy density.

The electrodes were then placed in 30 % KOH (prepared from mercury cell grade 45 % KOH) and charged against nickel dummies to a minimum of 50 % overcharge (based on the calculated theoretical capacity) during a 16-hour charge cycle. They were then discharged to 0.0 volts versus a nickel oxide reference electrode at a current of 200 mA, washed free of KOH in hot deionized water and air dried at 70° C for one hour.

D. ELECTROCHEMICAL TESTING

Cadmium electrodes thusly prepared were cycled in 2.35 x 1.62 in. sizes in specially constructed prismatic hardware in a nickel-cadmium-nickel sandwich configuration, using nickel electrodes of sufficient capacity to assure negative limited performance under all test conditions. These cells were cycled on fully automatic equipment to 100 % depth (0.00 volts) using the regime shown below:

<u>CYCLE NO.</u>	<u>CHARGE RATE</u>	<u>DISCHARGE RATE</u>
1-3	150 mA For 16 Hr	240 mA
4	150 mA For 16 Hr	1.20 A
5	150 mA For 16 Hr	2.40 A
6	150 mA For 16 Hr	4.80 A
7-End	1.20 A For 1 Hr	1.20 A

All the charge and discharge data were recorded on a Leeds & Northrup Speedomax Type G multipoint recorder and a statistically significant sample size was tested for each type of electrode prepared.

The use of negative limited cells assured that these electrodes will receive more tortuous treatment than they would receive in a practical cell. In this way, performance differences and aging characteristics between different type electrodes would show themselves with the expenditure of a minimum amount of test effort. Tests of longer duration, up to 250 cycles, performed on state-of-the-art electrodes showed no additional degradation beyond that attained in the first 56 cycles.

E. DATA REDUCTION

The large bulk of data collected during the characterizing and testing of the specimens of this study was reduced and correlated by use of a Honeywell Time-Sharing terminal and an array of standard and special computer routines that permitted rapid and accurate data processing.

III. RESULTS

A. BULK PROPERTIES OF ISOSTATICALLY COMPACTED PLAQUES

As briefly mentioned earlier, measurements of bulk plaque properties such as length, width, thickness, screen and sample weights were routinely made. In addition, powder weights, sample and sintered powder porosities, were calculated. These are summarized in tabular form below:

PROPERTY	$\bar{X} \pm 95\% \text{ CONFIDENCE LIMIT}$
Screen Weights*	$0.204 \pm 0.007 \text{ g/in.}^2$
Width	$1.629 \pm 0.005 \text{ in.}$
Length	$2.354 \pm 0.029 \text{ in.}$
Avg Thickness Variation In A Group**	$3.571 \pm 0.215\%$
Powder Content 60 % Specimens	$3.066 \pm 0.045 \text{ g/cc}$
Powder Content 80 % Specimens	$1.417 \pm 0.031 \text{ g/cc}$
Powder Porosity 80 % Specimens	$84.08 \pm 0.35\%$
Powder Porosity 60 % Specimens	$65.55 \pm 0.51\%$

* Did not follow normal distribution — perhaps 2 different batches

** Σ % variation within a group/n

n = number of groups

$$\% \text{ variation} = \frac{\text{Max. value} - \text{Min. value}}{\text{Max. value}} \times 100.0$$

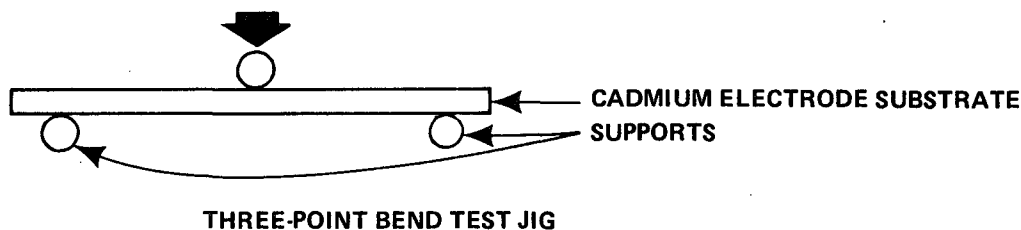
In spite of the fact that no rigorous effort was made to optimize uniformity on the bulk scale, a very high degree of uniformity is inherent in the process. Within the rather tight limits defined above, one would not expect the experimental results to be confounded with the above mentioned parameters.

A comparison of the uniformity of isostatically compacted plaques with those produced by other means has been made elsewhere².

B. MECHANICAL STRENGTHS OF ISOSTATICALLY COMPACTED PLAQUES

The mechanical strengths of cadmium electrode substrates are important properties to consider when one is preparing electrodes that are expected to be long lived. Furthermore, mechanical strengths are useful tools for general characterization of electrodes and plaques.

The samples whose mechanical strengths were to be evaluated were tested on a Instron three-point bend test jig shown schematically below:



The sample is supported by two rigidly mounted outer supports with a center-to-center distance of two inches. A third member is brought down to bear on the sample on its midpoint at a constant velocity. The sample is thusly slowly bent and eventually cracked. The load required to do this is obviously a measure of the mechanical strength of the electrode and should be a measure of the interparticle bonding of the plaque.

The load measurements are actually performed on an Instron Tensile Tester with the mobile pin attached to the crosshead of the instrument. The load required to crack the sample was determined with a one-pound load cell. Samples were cut to approximately one-inch widths, with the exact width being measured with a vernier at the fracture. Loads required to fracture the sample are usually in the 0.5 lb range.

An analysis of the bending moments gives for the yield stress, S , in units of psi:

$$S = \frac{3}{2} \frac{PL}{Wh^2}$$

Where:

P = load measured in pounds

L = sample length (two inches in this work)

W = sample width

h = sample thickness

Stresses expressed in this way were determined for randomly selected substrates.

The mechanical strengths are shown as a function of porosity in Figure 5. A least squares fit is given. The detailed data are in Appendix A.

The values of the mechanical strengths are seen to vary linearly between 900 and about 8000 psi between porosities of 80 and 60 %. A variation in mechanical strengths with induced pore size was noted with the larger pore-size materials having in general higher strengths. The differences in mechanical properties with varying induced pore-size was extremely small and will not be elaborated on at this particular time. The mechanical strength is shown to be a rapidly varying function of porosity; much greater than one might anticipate if only the metal content of the plaque were considered.

Especially noteworthy are the exceptionally high values of mechanical strength of the 60 % plaques, in the 8000 psi range. Conventional gravity sintered plaques sintered under the same conditions had mechanical strengths of 643 for a 79.2 % porous plaque. If the performance of electrodes prepared from the compacted 60 % plaques is on a par with the gravity sintered materials, important benefits, like life for example, may be realized.

C. B.E.T. SURFACE AREAS OF SUBSTRATES

Surface areas of special specimens were determined using a modified B.E.T. method and Krypton as the adsorbate. The surface areas were in the range of 0.11 to 0.17 m²/gm of powder content in the plaque. The contribution of the screen weight to the total plaque weight was arithmetically eliminated for the sake of this discussion.

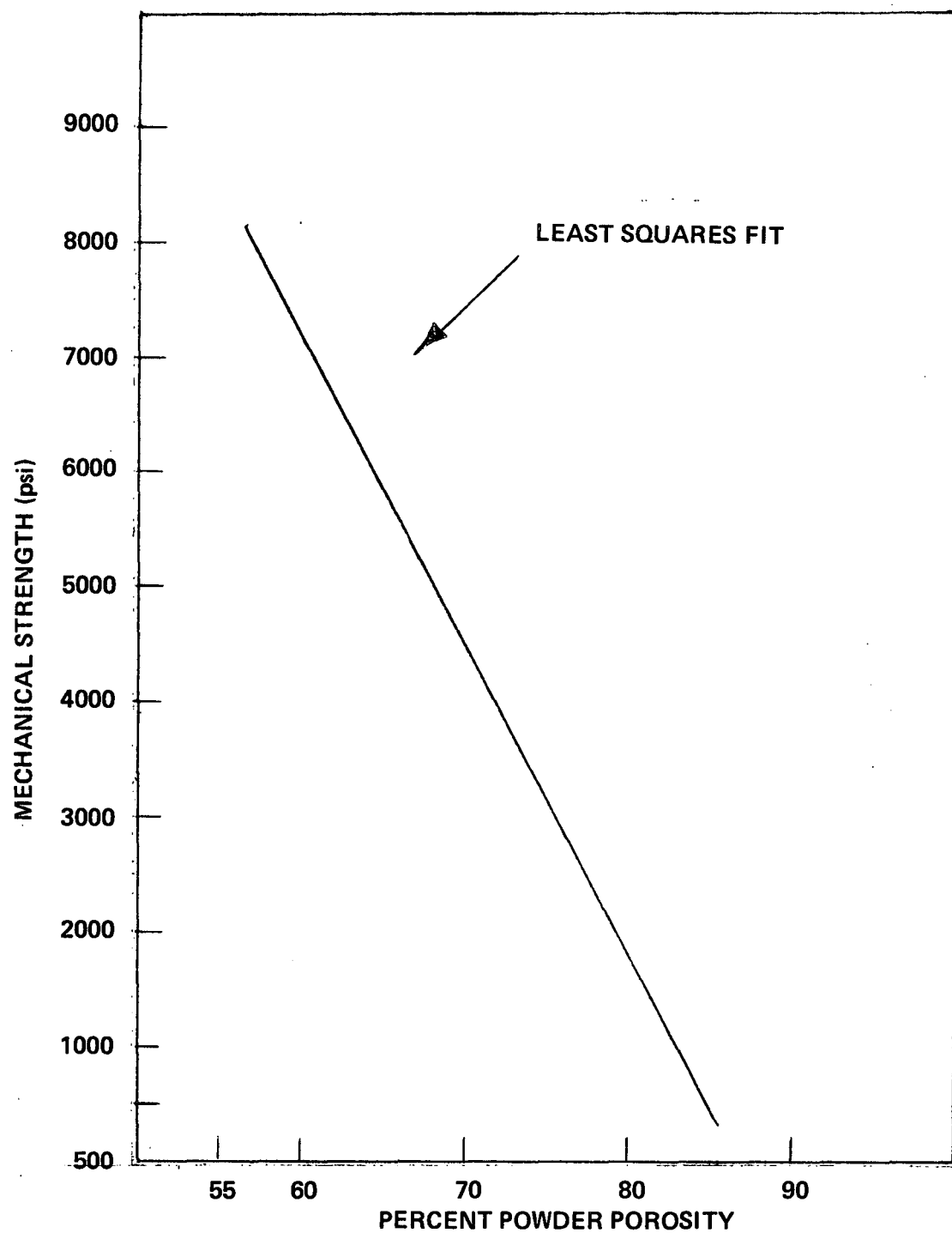


FIGURE 5. RELATION BETWEEN MECHANICAL STRENGTH AND POROSITY

The specimens whose surface area was measured did have support screens however. The specific surface areas in terms of powder weights are shown as a function of total porosity including screen in Figure 6. The 87 % porosity materials were studied previously in a privately funded project. The specific surface area is seen to be a linear function of porosity in this range for the two different pore sizes. The reduction in surface area with decreasing porosity is a reflection of the packing of the nickel powder. With the low porosity materials a particle has more neighbors to bond with during pressing and form necks with during sintering. There is, therefore, a marked decrease in powder surface area with decreasing porosity.

The surface areas of the compacted plaques also show some dependence on induced pore-size. This effect is rather small however. One may easily visualize changes in the number of interparticle bonds and therefore, surface area when the size and quantity of pore-former particles are varied. The use of larger pore-former particles with the same quantity of metal powder results in a material with a thicker pore wall. The additional powder around the pore-former seemingly packs differently for different sized pore-formers. As a result each particle will have a different number of neighbors to form necks with during sintering depending upon the size of the pore-former. The surface area will, therefore, exhibit small differences.

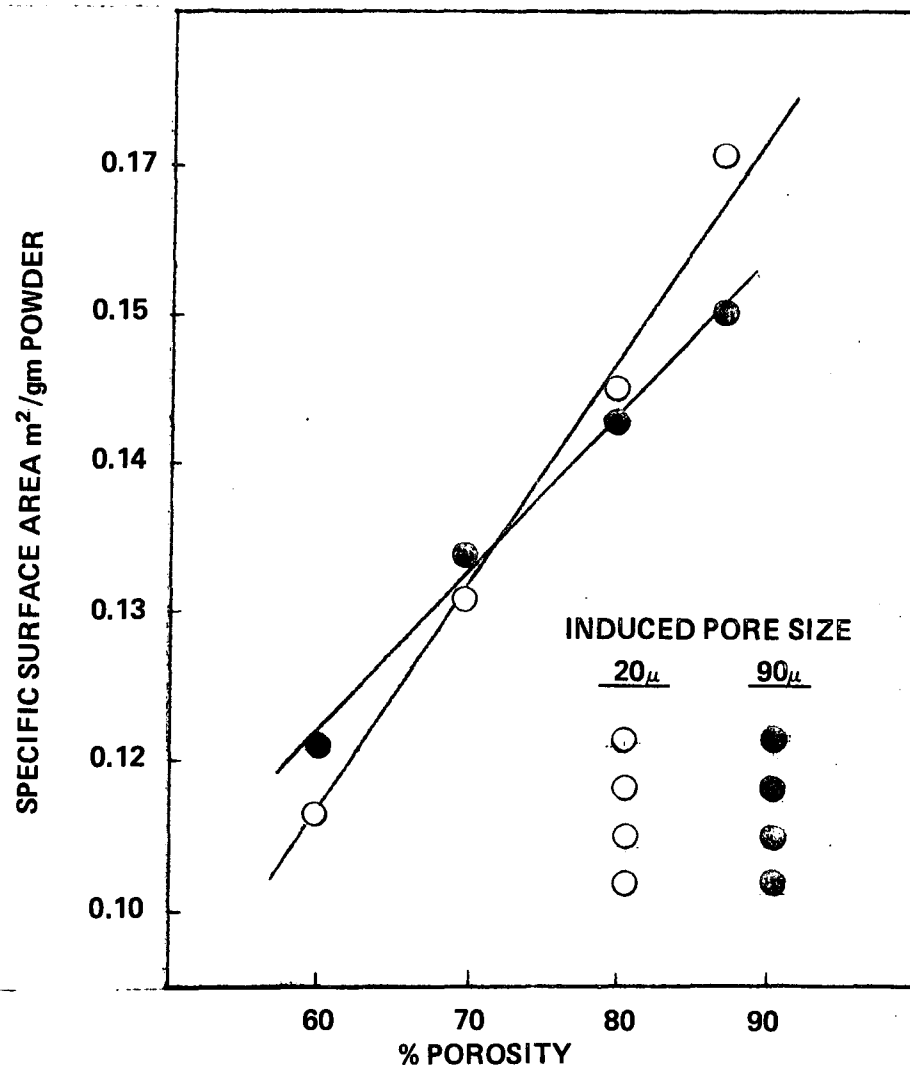


FIGURE 6. SPECIFIC SURFACE AREA AS A FUNCTION OF PLAQUE POROSITY

Figure 6 shows the two lines intersecting. On the basis of the above discussion, one would not expect this to happen. In reality, different ratios of pore-former to nickel powder are required to prepare plaques of differing porosity irrespective of the pore-size. One would expect that where the results are anomalous, the same ratio of pore-former to nickel powder ratio was not employed.

D. RESULTS OF CYCLE TESTING

As described earlier, accelerated life testing of the cadmium electrodes prepared was performed in specially constructed negative limited cells at high charge and discharge rates. This is an extremely punishing regime for cadmium electrodes. It was employed in an effort to minimize the amount of experimental effort in evaluating the differences between various electrodes. The results are therefore not necessarily typical of cadmium electrodes in conventional positive limited systems in which they are never discharged to 100 % depth. The results are therefore best used only for comparative study of this type. Testing of 80 % porosity plaques of 90, 50, 25, and 15 μ induced pore size and 60 % porosity plaques at the 90, 50, 25, and 15 μ induced pore-size levels has been completed. Also, performance of state-of-the-art electrodes (gravity sintered plaques) were evaluated for the purpose of comparison. In addition, a blended powder, Inco 287 with Sheritt-Gordon 1 μ powder, was evaluated.

Test results for the state-of-the-art electrodes are given in Figure 7. Cycle by cycle data is given in Appendix B. The points are averages of eight cells and the error bars represent the 95 % confidence interval from the average. We are now in a position to evaluate test results of our new plaques on a comparative basis.

1. Test Results For 80 % Porous Plaques

Accelerated cycle test results for cadmium electrodes prepared from isostatically compacted plaques of 80 % porosity and 15, 25, 50, and 90 μ induced pore size are shown in Figures 8 through 11, respectively. Cycle by cycle data is given in Appendix B. Periodic oscillations in the test data were observed at times as for example in Figure 7. We have no explanation for this observation. No outstanding differences between these groups of cells was evident. There were some differences however. The group of electrodes prepared from the 80 % 25 μ pore-size plaques had higher efficiencies than the other groups of cells, especially during later stages of the test program. Electrodes prepared from the plaques with 50 μ and 90 μ induced pore-sizes, although they were slightly lower in efficiency, were more uniform in performance. This difference was due to the ease of handling and blending pore-former particles with nickel particles, which manifested itself in better general plaque uniformity (see following sections). Special difficulties were encountered with the preparation and blending of the 15 μ pore-former.

It thus appears that there is little or no contribution of induced pore-size to electrode efficiency at these porosity levels. And more significantly, all these groups of cells are comparable to the state-of-the-art electrodes. The electrodes prepared from the 80 % 25 μ plaques were better throughout the test.

Cycle test data were generated for an 80 % porous plaque containing 60 % Inco 287 and 40 % Sheritt-Gordon 1 μ nickel powder. The test results are given in Figure 12 and are comparable to the 80 % 25 μ electrodes, that is, there is no statistically significant difference between them.

2. Test Results For 60 % Porous Plaques

Accelerated cycle testing was also completed for cadmium electrodes prepared from 60 % plaques at the 15, 25, 50, and 90 μ pore-size levels. Test results are given in Figure 13 through 16, respectively. The electrodes prepared from the 25 μ induced pore plaques had the best efficiencies of the group. The 60 % 25 μ electrodes

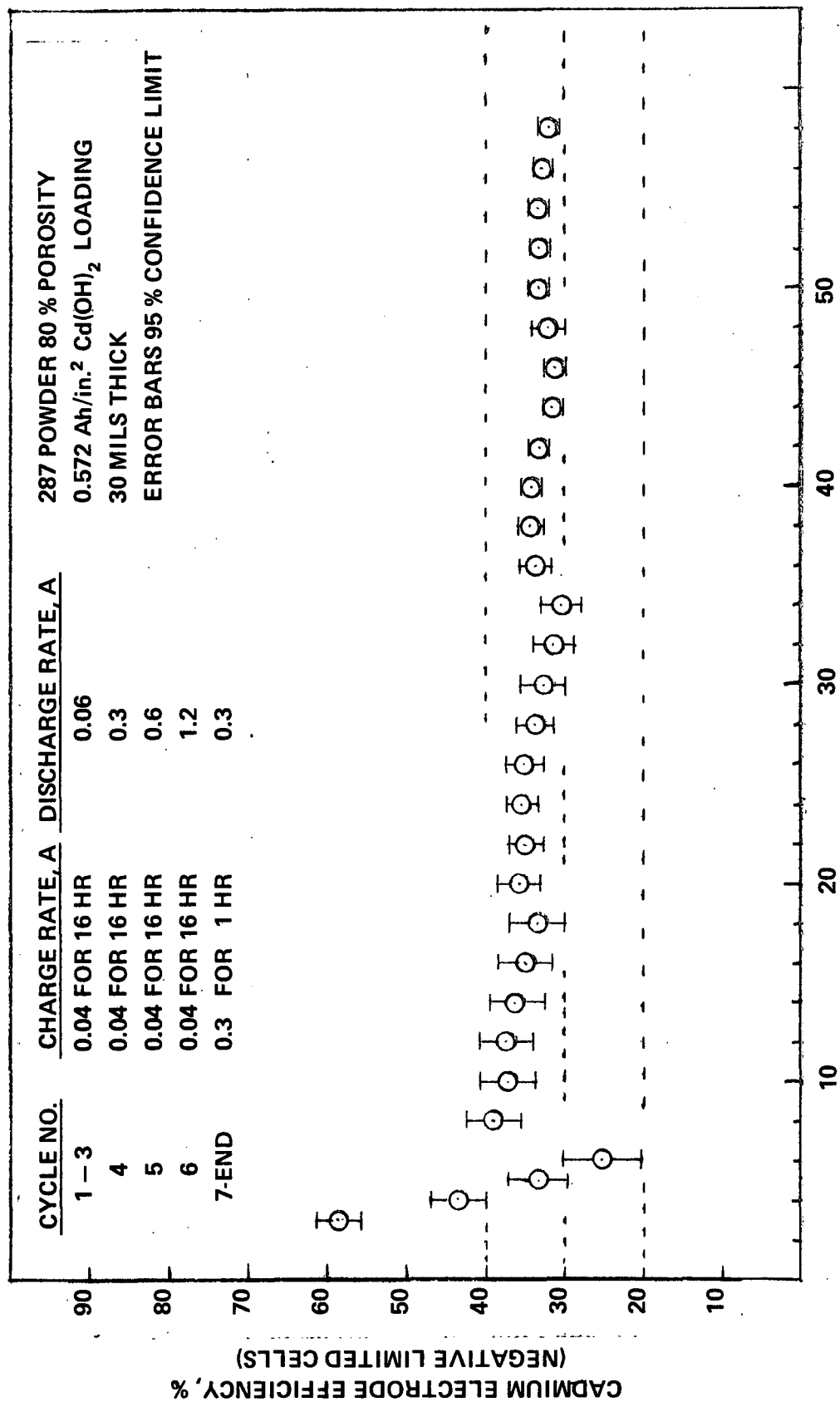


FIGURE 7. CYCLE LIFE OF STATE-OF-THE-ART CADMIUM ELECTRODE

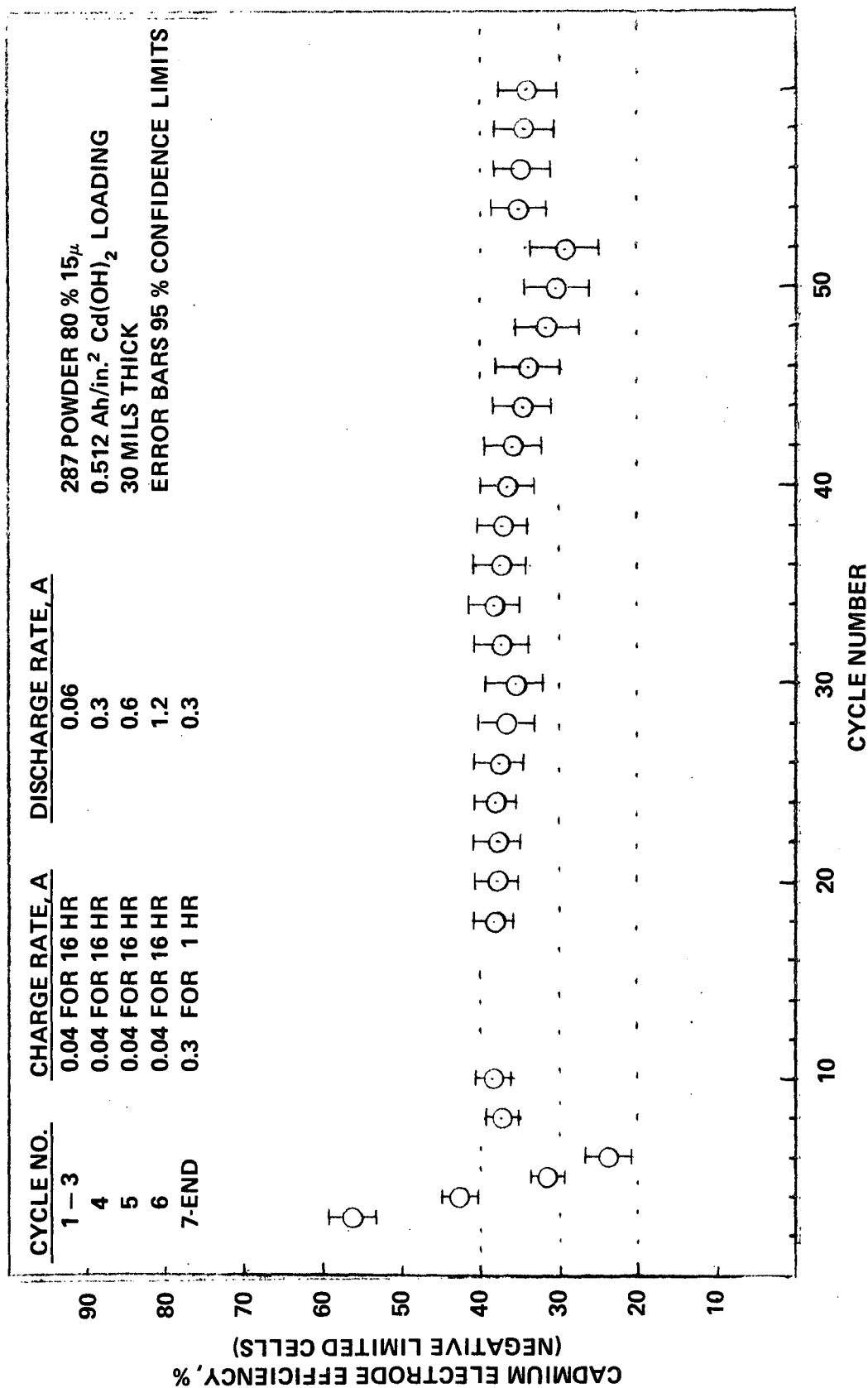


FIGURE 8. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES 80 % POROSITY 15μ INDUCED PORES

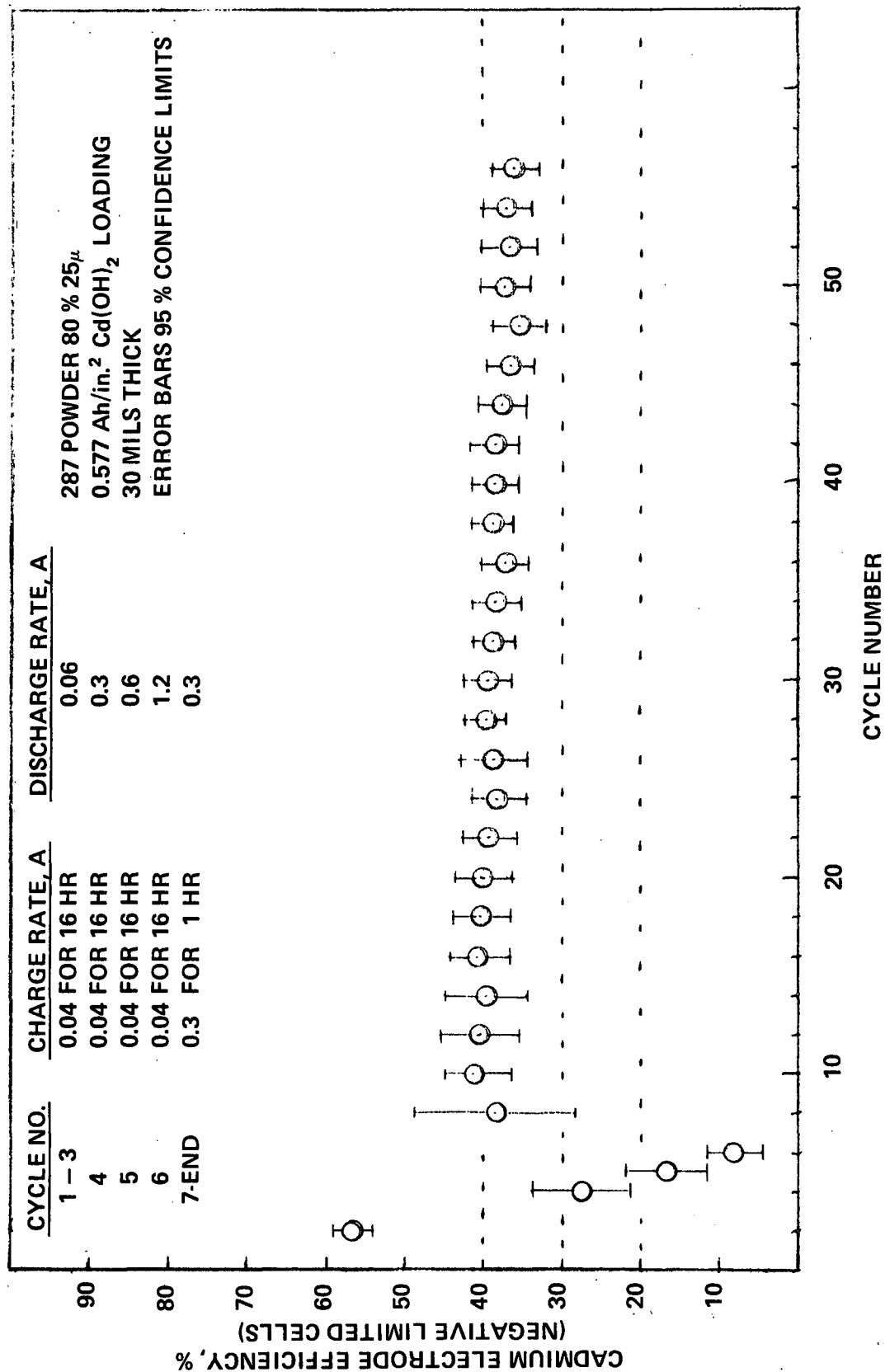


FIGURE 9. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 80 % POROSITY 25μ INDUCED PORES

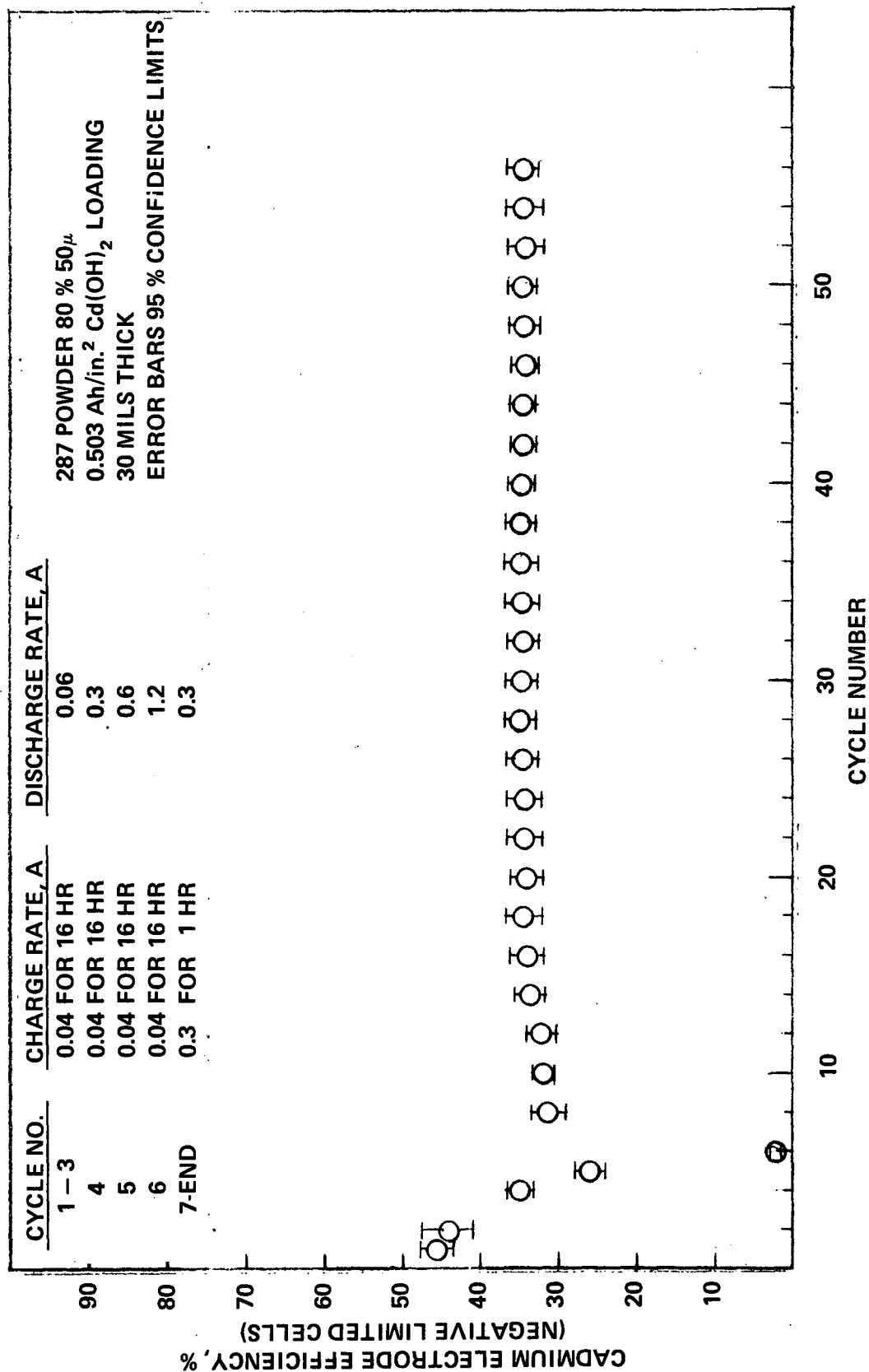


FIGURE 10. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES 80 % POROSITY 50μ INDUCED PORES

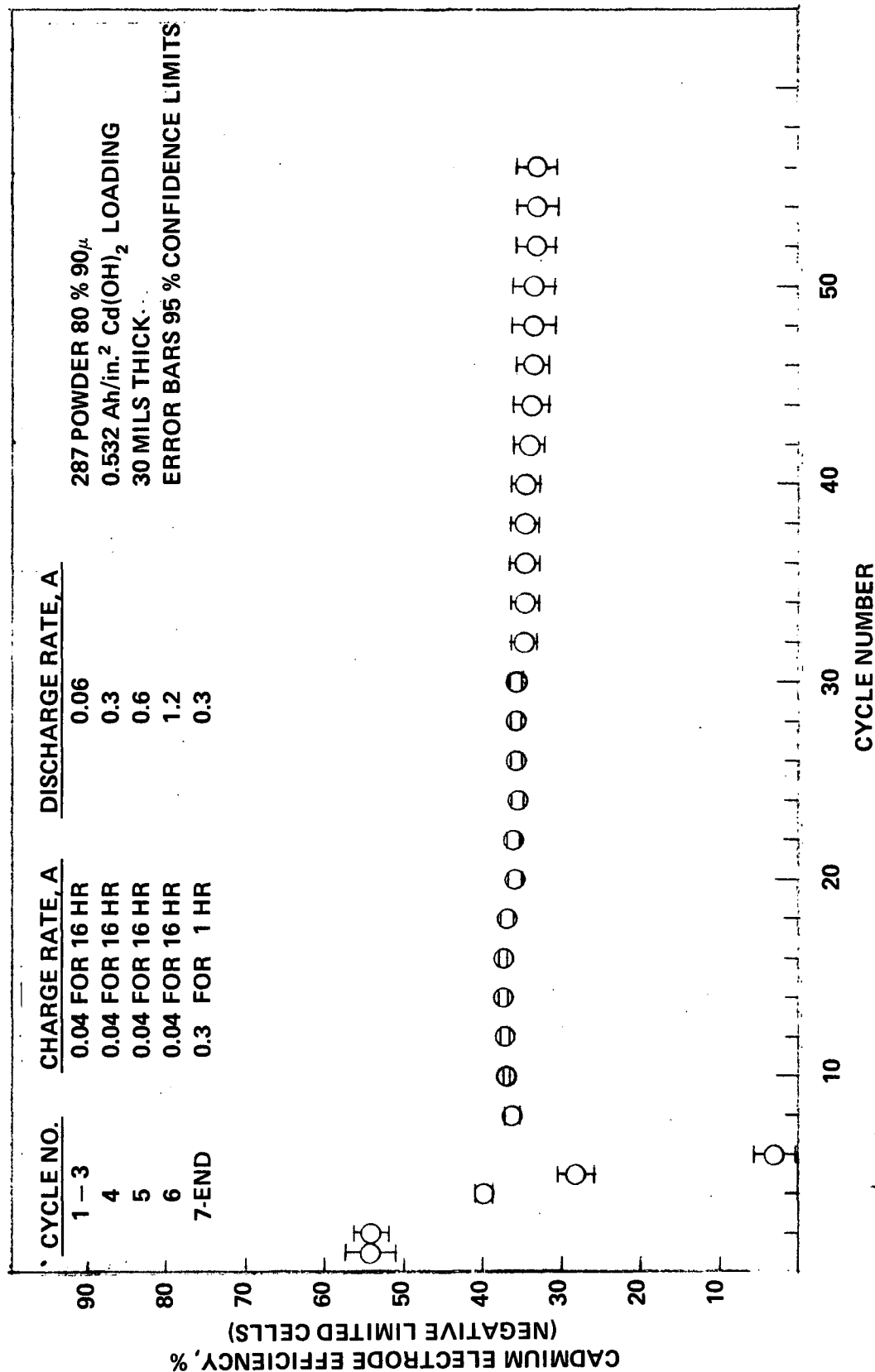


FIGURE 11. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES 80 % POROSITY 90μ INDUCED PORES

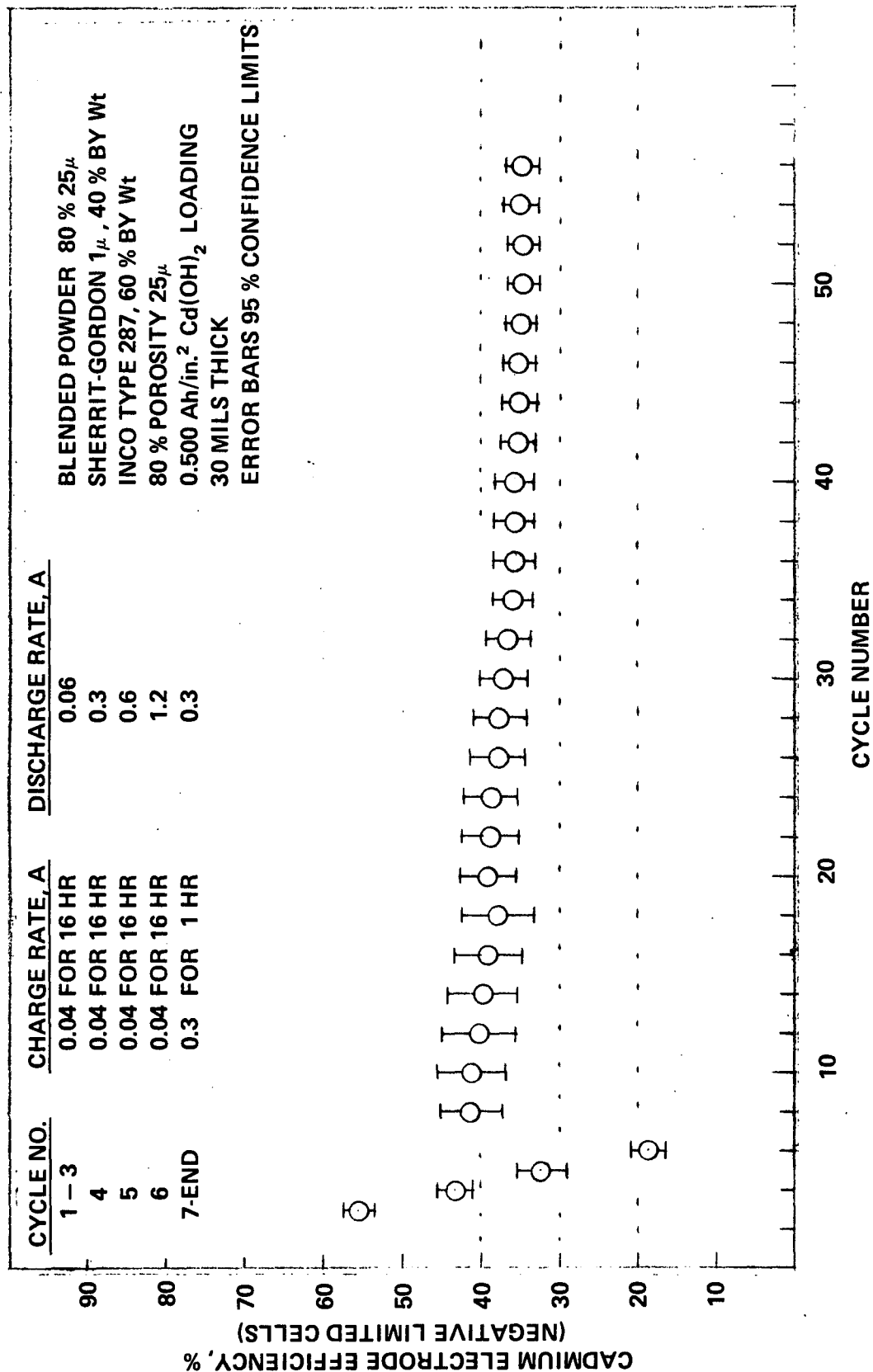


FIGURE 12. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 80 % POROSITY
 25 μ INDUCED PORES BLENDED POWDER

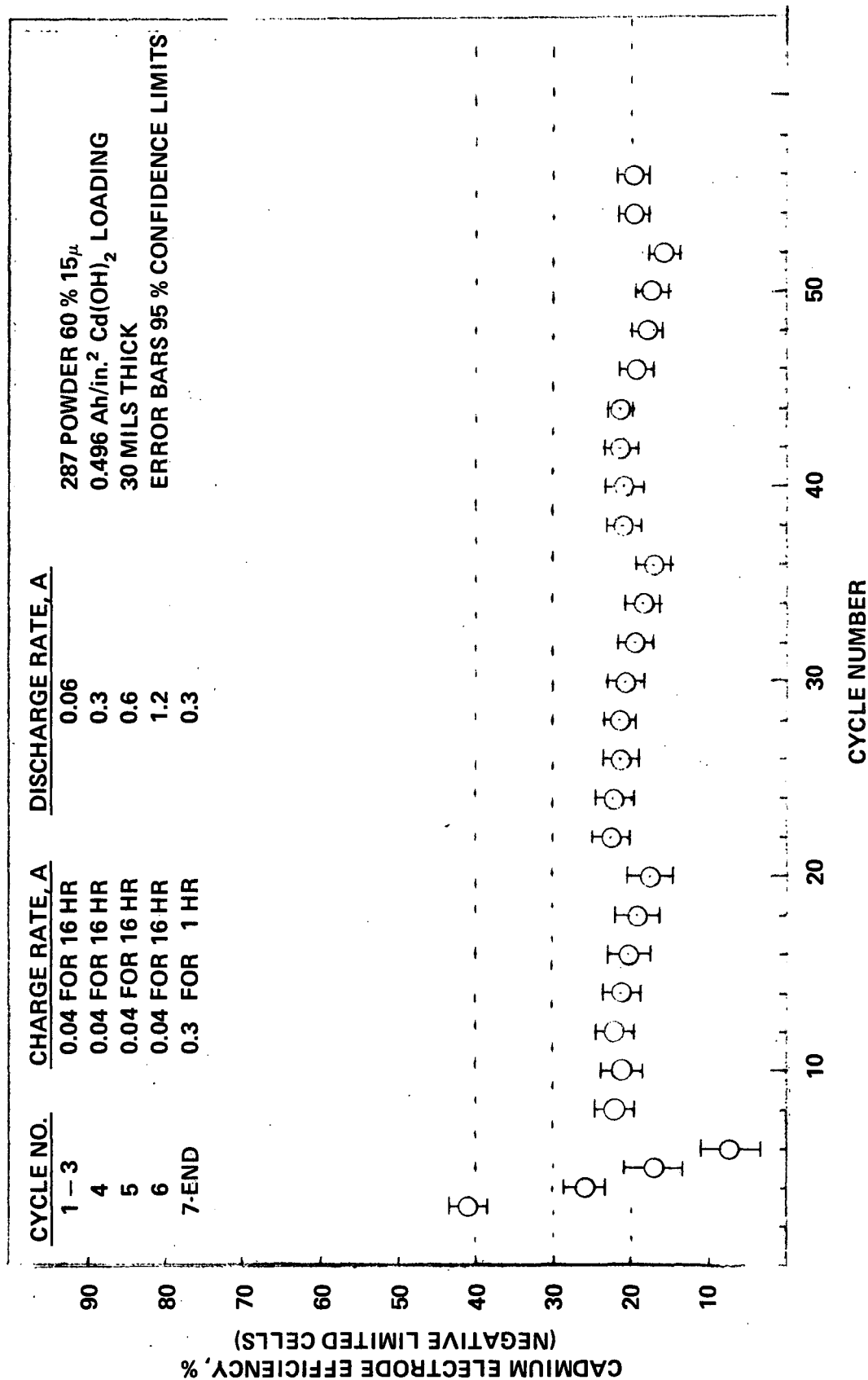


FIGURE 13. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 60 % POROSITY 15μ INDUCED PORES

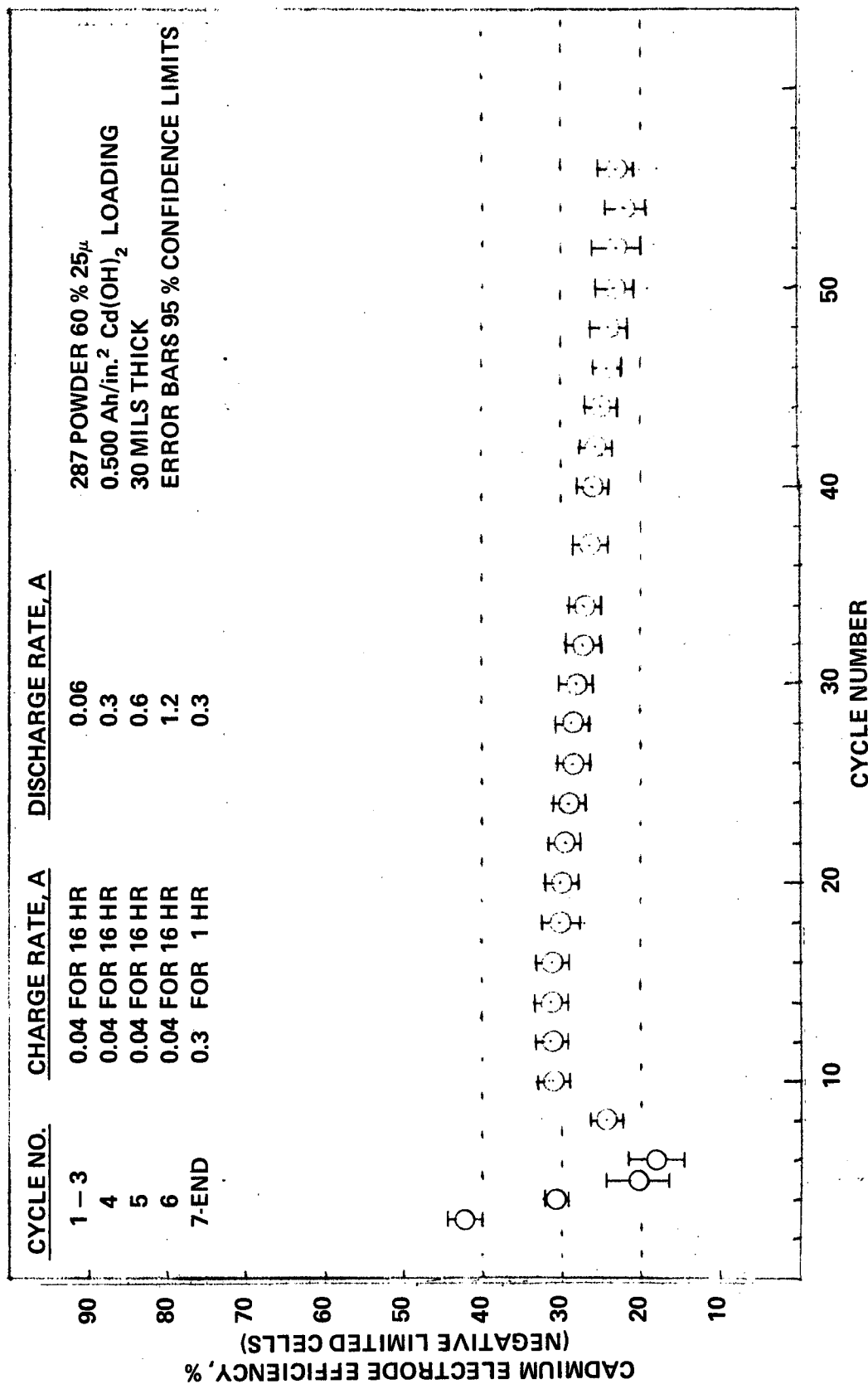


FIGURE 14. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 60 % POROSITY 25μ INDUCED PORES

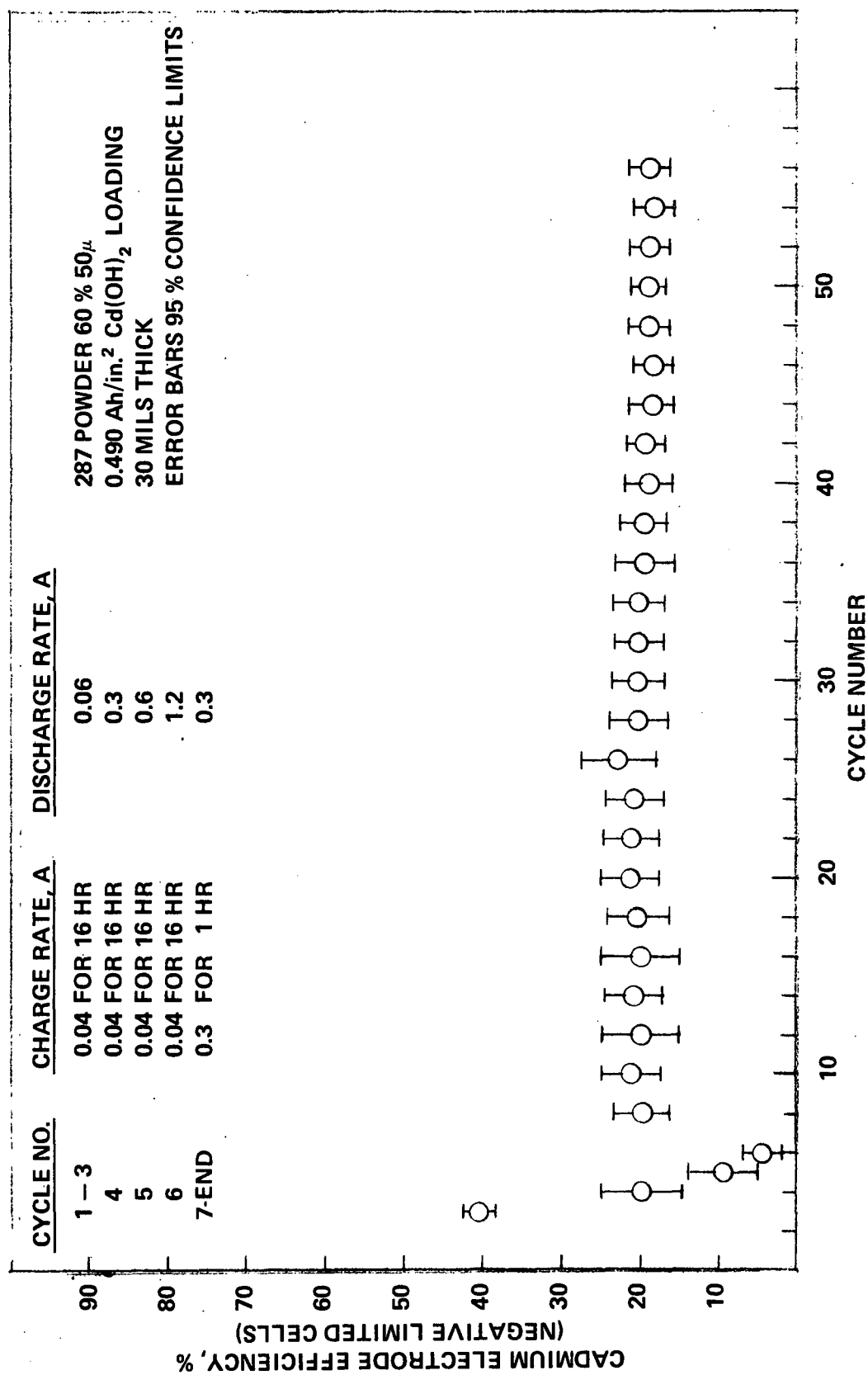


FIGURE 15. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 60 % POROSITY 50μ INDUCED PORES

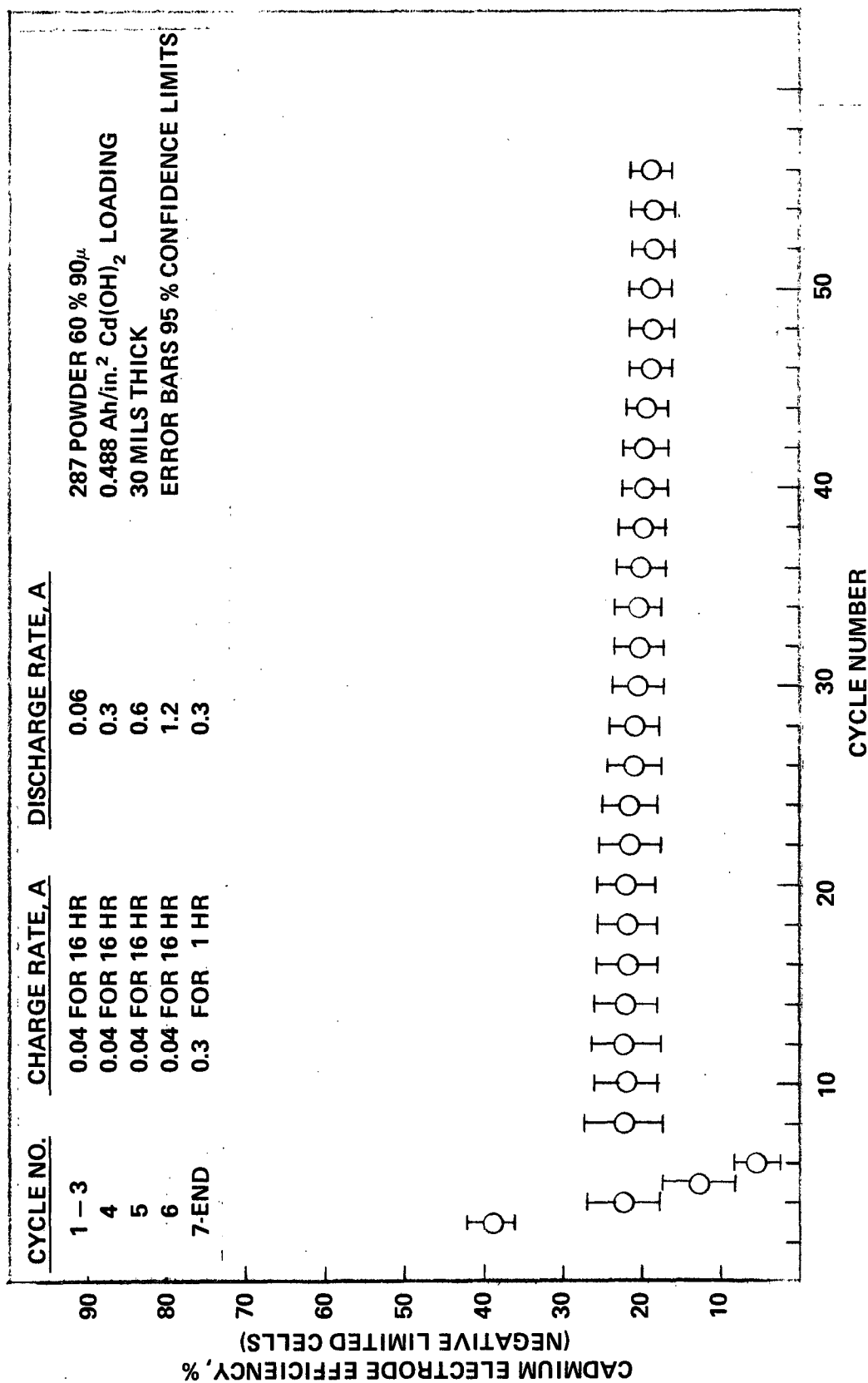


FIGURE 16. CYCLE LIFE OF CADMIUM ELECTRODES PREPARED FROM
 ISOSTATICALLY COMPACTED PLAQUES 60 % POROSITY 90μ INDUCED PORES

are however, lower in efficiency than the 80 % electrodes but not by a great deal, about 10 % across the board. In view of their exceptional mechanical properties, as compared to the 80 % structures (see Figure 5) and good efficiency and with the prospect of improving the efficiency, these are extremely interesting and promising structures, in the opinion of the authors.

3. Test Summaries

A summary of the test results of the accelerated cycle data obtained to date is given in Table 1. The degradation of capacity rates was determined via a linear regression analysis of the test data, using the seventh to the last cycle. A straight line was found to fit the data well. The differences in the test data were small, a numerical summary makes it easier to compare the isostatically compacted electrodes and conventional electrodes prepared from gravity sintered plaques from the point of view of efficiency and degradation rates. As far as degradation rates are concerned there are no statistically significant differences, with one exception, between all the electrodes tested. The electrodes prepared from the 60 % 25 μ plaques inexplicably have higher degradation rates than all the other electrodes. It therefore seems that the degradation rate under the test conditions employed is independent of the plaque properties and therefore can not be used as a criterion for cell life. If there is any distinguishing feature in the data it would have to be the electrode efficiencies where some real differences are observed. From this point of view the 80 % structures with pore sizes 25 or less have efficiencies higher than conventional electrodes.

It is seen that electrodes prepared from the higher induced porosities, 91 % versus 49 %, have considerably higher efficiencies at the end of 50 tortuous cycles. We cannot account for this difference on the basis of increased loading per unit volume of the free space of the 60 % electrodes. (Recall that all electrodes had the same loading per unit total volume.) On the basis of their loadings alone, we expected 60 and 80 % porous electrodes to have essentially the same efficiencies. The low efficiencies of the 60 % electrodes are perhaps related to their low induced porosity. Further work will be required if this phenomenon is to be clarified.

TABLE 1. SUMMARY OF CYCLE TEST DATA

Least Squares Fit To $Y = A + BX$

Y = Efficiency, X = Cycle Number

ELECTRODES	% P _{ind} ⁽¹⁾	A ⁽²⁾	B ⁽³⁾	SMOOTHED EFFICIENCY AT CYCLE 50
State-of-the-Art (Thermal)	0	37.33	-0.100 \pm 0.058	32.3 \pm 2.4
From 60 % 15 Plaques	40	22.22	-0.0694 \pm 0.054	18.7 \pm 2.3
From 60 % 25 Plaques	40	33.20	-0.182 \pm 0.052	24.1 \pm 2.2
From 60 % 50 Plaques	40	21.98	-0.064 \pm 0.086	18.8 \pm 3.6
From 60 % 90 Plaques	40	23.22	-0.085 \pm 0.082	19.0 \pm 3.5
From 80 % 15 Plaques	91	40.28	-0.120 \pm 0.076	34.3 \pm 3.2
From 80 % 25 Plaques	91	41.51	-0.0873 \pm 0.092	37.1 \pm 3.9
From 80 % 25 Plaques	91	41.64	-0.140 \pm 0.079	34.6 \pm 3.3
(Blended Powders)				
From 80 % 50 Plaques	91	32.54	+0.048 \pm 0.048	34.9 \pm 2.0
From 80 % 90 Plaques	91	38.00	-0.086 \pm 0.031	33.7 \pm 1.3

(1) Percent Porosity Induced, calculated

(2) Electrode Efficiency at the Zero Cycle, if it is possible to extrapolate through the first 7 cycles \pm average 95 % confidence limit

(3) Rate of degradation of the electrode efficiency per cycle \pm 95% confidence limit

E. SCANNING ELECTRON MICROSCOPY OF ISOSTATICALLY COMPACTED PLAQUES AND CADMIUM ELECTRODES

The scanning electron microscope (SEM) is a uniquely suited tool for examining the extent of uniformity of the microstructures of the plaque materials prepared in this work. This is mainly due to the striking superiority of the SEM micrographs as compared to optical micrographs. This important benefit is in addition to other very considerable advantages such as:

- Not requiring special and lengthy polishing of a flat surface which is difficult for soft battery plate materials we are dealing with.
- No need to etch the specimen to differentiate the different materials and phases present.
- Surfaces do not have to be replicated as is required in conventional electron microscopy.

The utility of the SEM will be evident as the micrographs are presented.

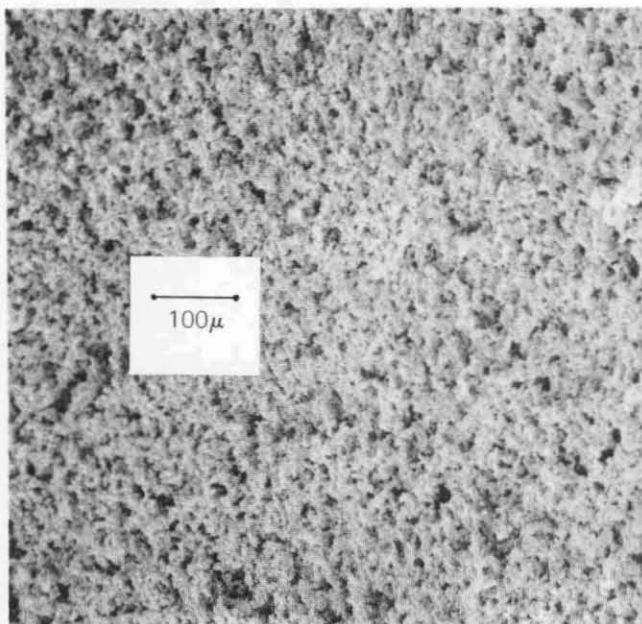
Aside from qualitatively judging the extent of uniformity, attempts were made to measure the total porosity and interstitial porosity from the photomicrographs.

It was not possible to determine these properties with any degree of accuracy from the SEM photomicrographs due to its extreme depth of field. It was not possible to identify all the pores since one could not readily distinguish nickel particles at different planes (levels) in the plaque. However, it was a relatively simple matter to determine the average induced pore size by measuring a representative sampling of the induced pores at the 100X magnification, of the micrographs given in the following text and in Appendix D. Results are given below in Table 2. These measurements are very sensitive to the judgement of the observer.

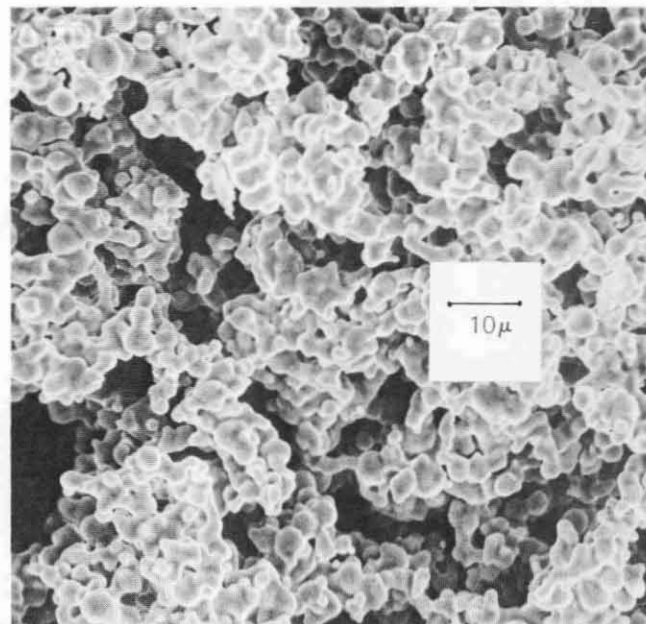
TABLE 2. INDUCED PORE SIZE OF ISOSTATICALLY COMPACTED PLAQUES

<u>PLAQUE</u>	<u>INDUCED PORE SIZE, μ</u>
60% 15 μ	10-15
80% 15 μ	10-15
60% 25 μ	16
80% 25 μ	16
60% 50 μ	33
80% 50 μ	35
60% 90 μ	72
80% 90 μ	70

Figures 17 and 18 are representative SEM photomicrographs, 60 and 80 % 25 μ , of isostatically compacted structures and are qualitatively quite revealing as far as microscopic uniformity is concerned. Part (a) of each figure at approximately 100X magnification shows the nature of the induced porosity, rather large pores (measured values given in Table 2 for all groups of plaques prepared), uniformly distributed throughout the plaque. The 80 % porosity material apparently being a little bit more uniform in this respect. The actual size of the induced pores are considerably less than sizes of the pore-former particles. The reduction in size is probably due for the most part to shrinkage during sintering. The photomicrographs at the 1000X magnification show the induced and interstitial porosity in more detail, especially the interstitial porosity and its relation to the induced

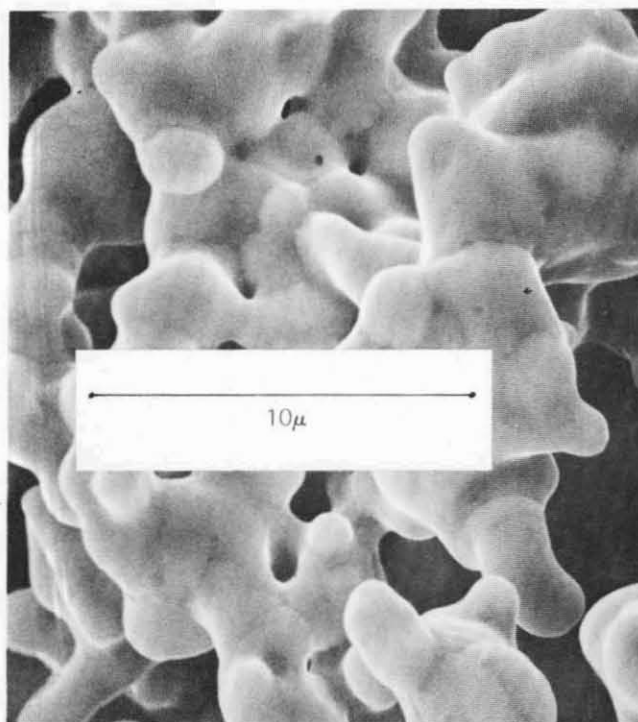


110X



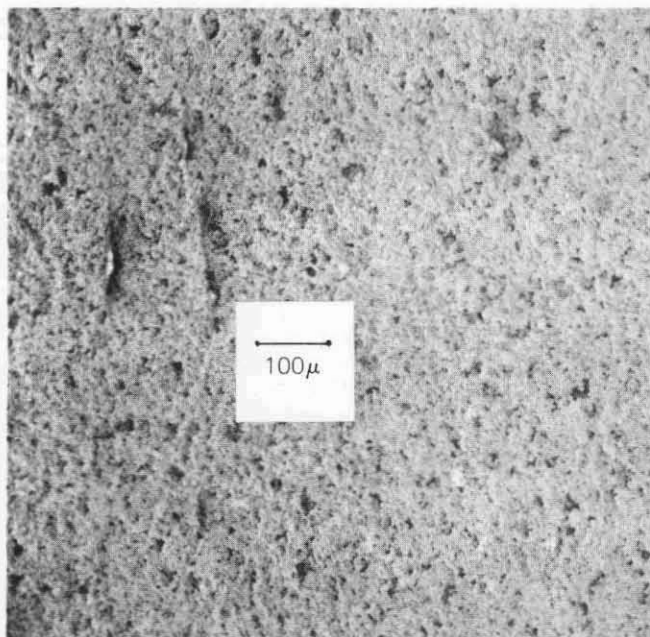
1000X

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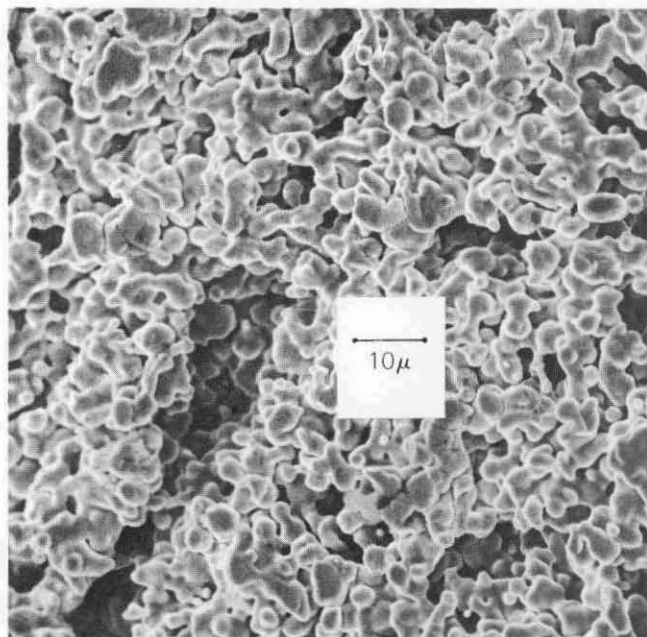


5000X

FIGURE 17. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY COMPACTED
PLAQUES, 80 % 25 μ

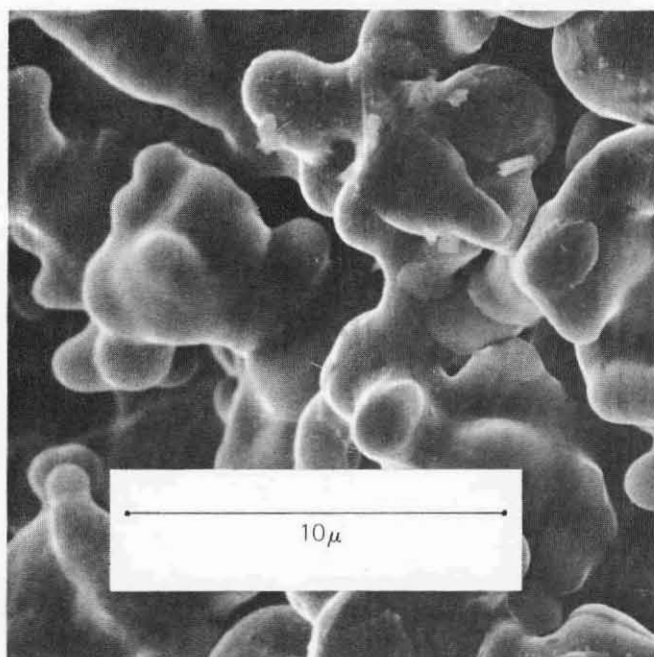


95X



1000X

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5000X

FIGURE 18. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUES, 60 % 25μ

porosity. Small interstitial pores well under 10μ are evident which no doubt contribute substantially to the total porosity and performance of the electrode. No very extensive agglomerates of material are visible, that is, all the porosity is seemingly interconnected. Part (c) at 5000X magnification reveals some interesting fine points about the sintered nickel structures. These show the interstitial porosity exclusively. The small interstitial pores are again evident. Also, linear boundaries are apparent. These represent grooves from the sintering of two nickel powder particles. Also evident are dots on the nickel structures. These are micro pits where three or more particles were sintered together. There is a far greater population of these dots for the compacted 80 % porous structures. This is indicative of more interparticle bonding and no doubt contributes to the increased strengths of the compacted materials over the conventional gravity sintered materials.

These observations are contrasted with the details shown in Figure 19 for a state-of-the-art gravity sintered plaque prepared under identical conditions of sintering time and temperature. Very large irregular pores not uniformly distributed are evident. Also present in the gravity sintered structure are islands of large agglomerates of nickel metal. The gravity sintered specimens do not have the well-developed necks and the high population of dimples as the compacted specimens.

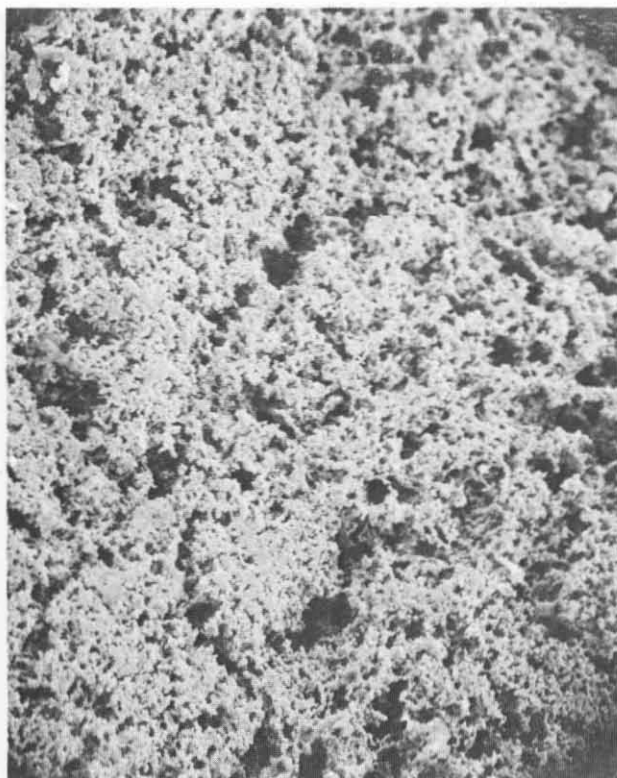
The compacted plaques characteristically have well-developed necks. The increased and improved interparticle contact between powder particles and the compressed plaques is the primary factor governing their improved strength.

Routinely at the end of the accelerated test program, SEM photomicrographs of selected specimens of the cycled cadmium electrodes were taken in an effort to measure morphological changes that occurred during the cycle testing. Typical examples are shown in Figure 20 and 21 for electrodes made from 60 % 25μ and 80 % 15μ plaques. The $\text{Cd}(\text{OH})_2$ crystal size through the plaque structure is important because the crystal size is perhaps a key property governing the performance degradation of cadmium electrodes during cycling.

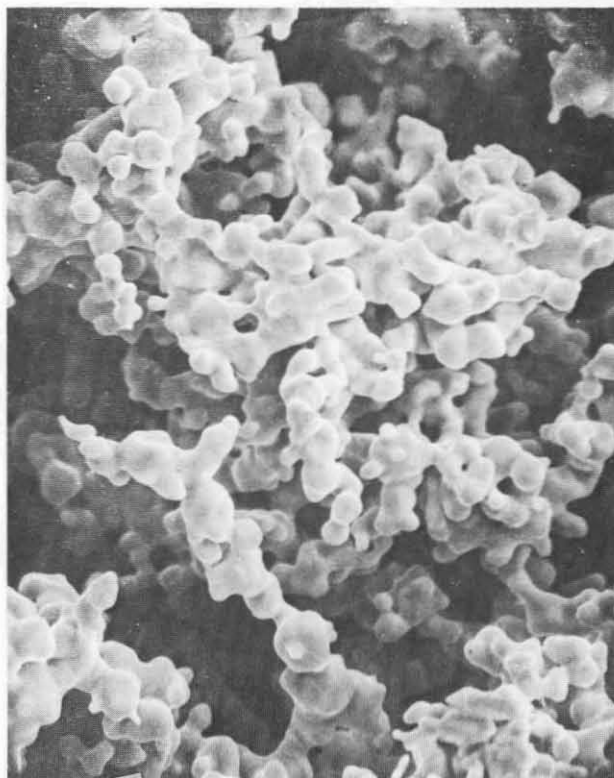
Also of importance in the micrographs is the presence of base nickel. A typical example of a formed but not cycled electrode is given in Figure 22. Note that the plaque substructure is completely covered with $\text{Cd}(\text{OH})_2$ crystals. Apparently with prolonged cycling, morphological changes occurred in which cadmium active mass migrated from the nickel surfaces onto other cadmium hydroxide particles. Also prominent in the micrographs is the evidence of corrosion of the nickel substrate. The previously discussed pits are considerably enlarged after cycling. At present we do not know whether this is due to electrode processing or to the cycling.

Additional SEM photomicrographs of isostatically compacted plaques (60 and 80 %, $15\text{-}90\mu$) and electrodes made from same are included in Appendix D and E.

The $\text{Cd}(\text{OH})_2$ crystal sizes, \bar{d} , were measured from the micrographs and are summarized below in Table 3 with other pertinent data, the smoothed 50th cycle efficiency.

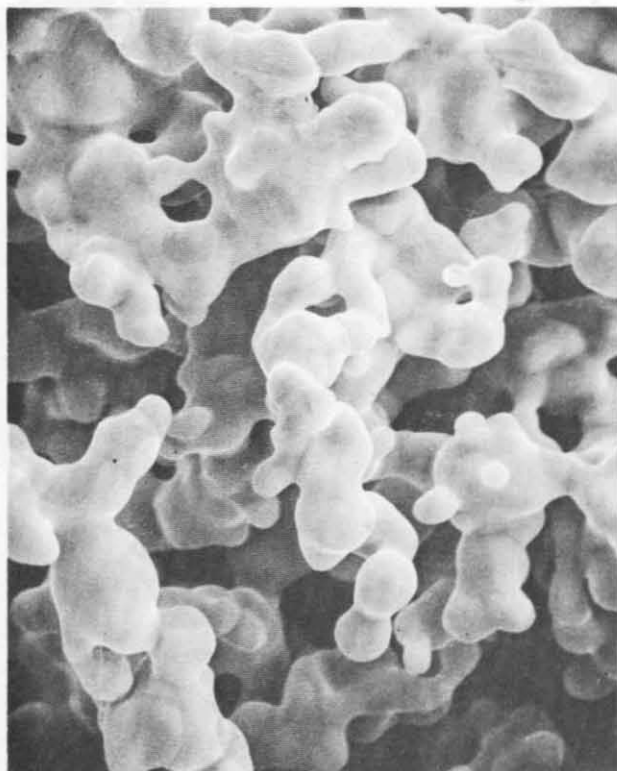


135X

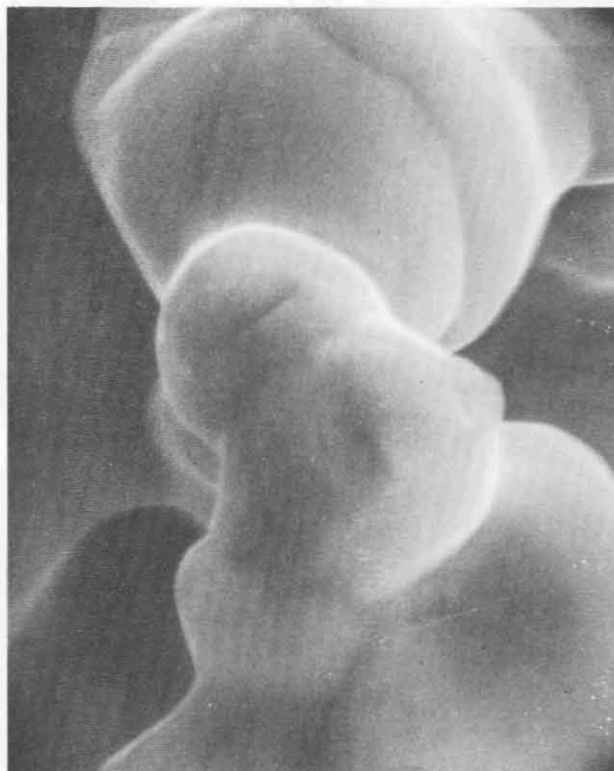


1350X

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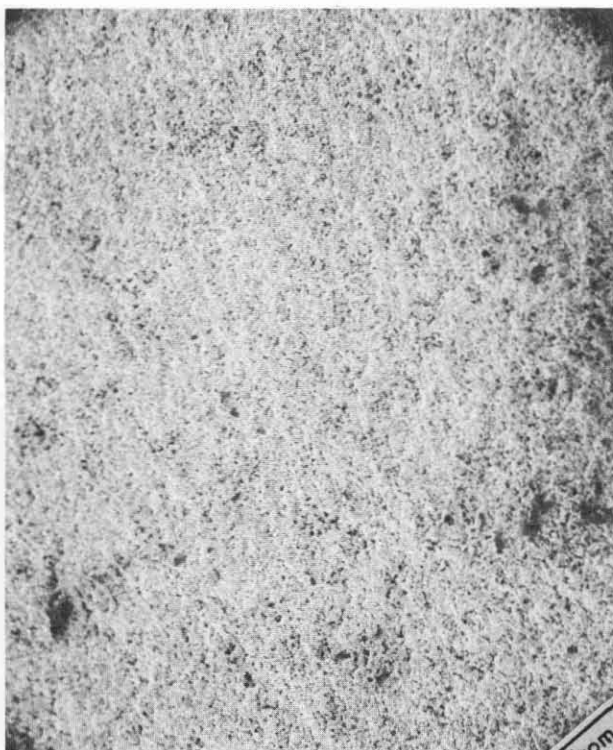


2305X

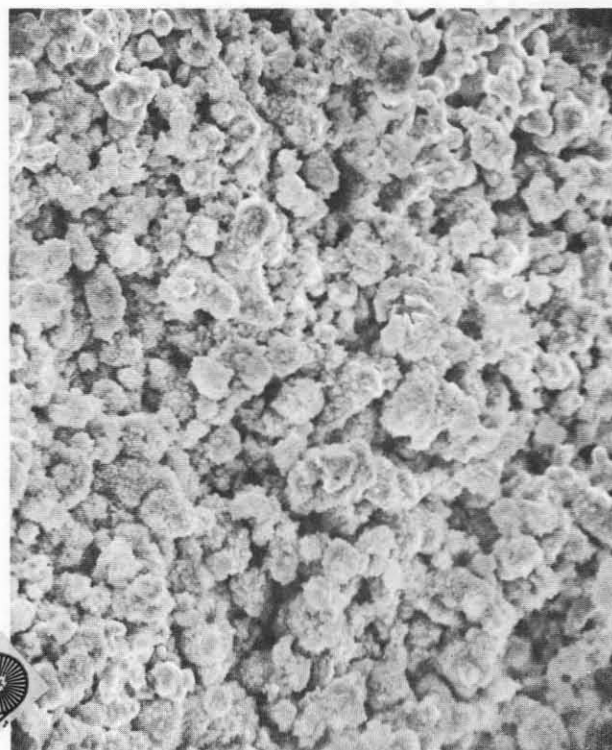


13,520X

FIGURE 19. SEM PHOTOMICROGRAPHS OF STATE-OF-THE-ART
GRAVITY SINTERED PLAQUES

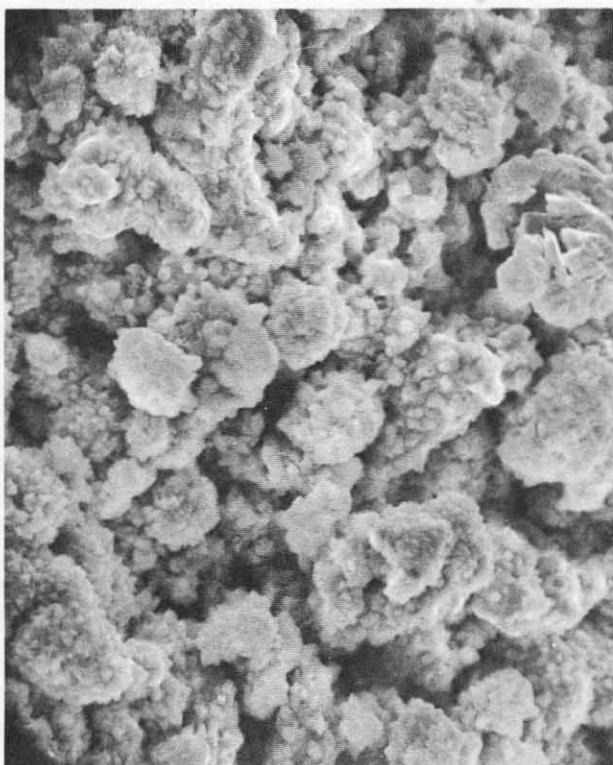


105X



1050X

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2100X

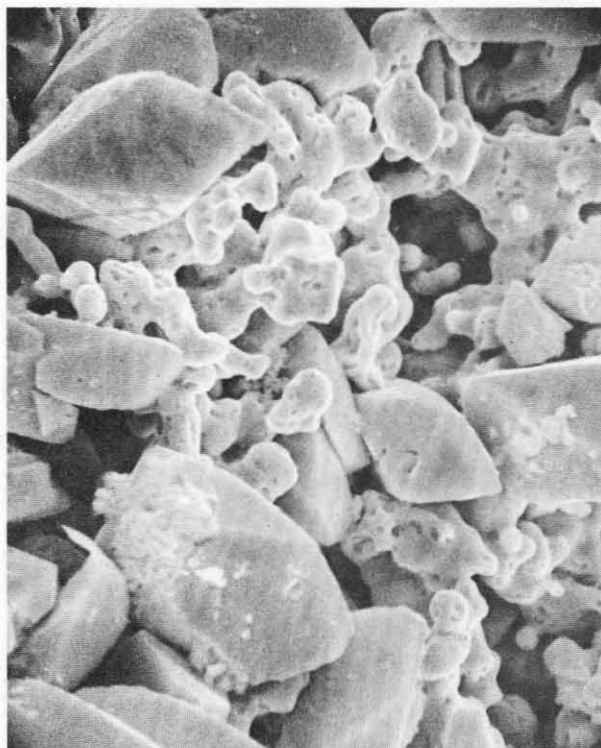


10,500X

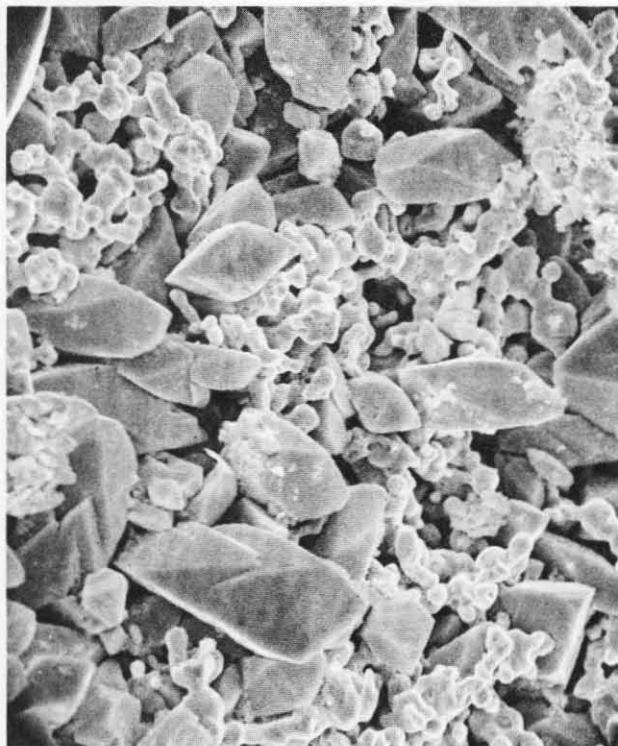
FIGURE 20. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODES MADE FROM
ISOSTATICALLY COMPACTED PLAQUES, 60 % 25 μ .



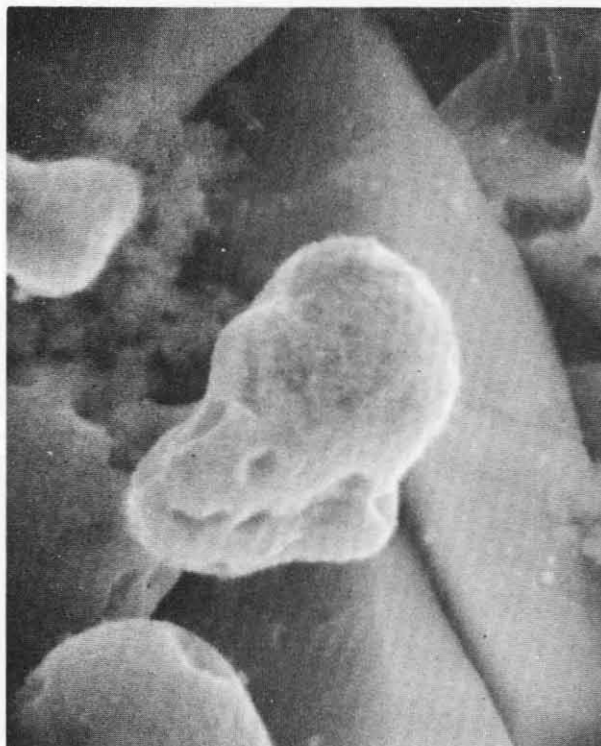
105X



1050X

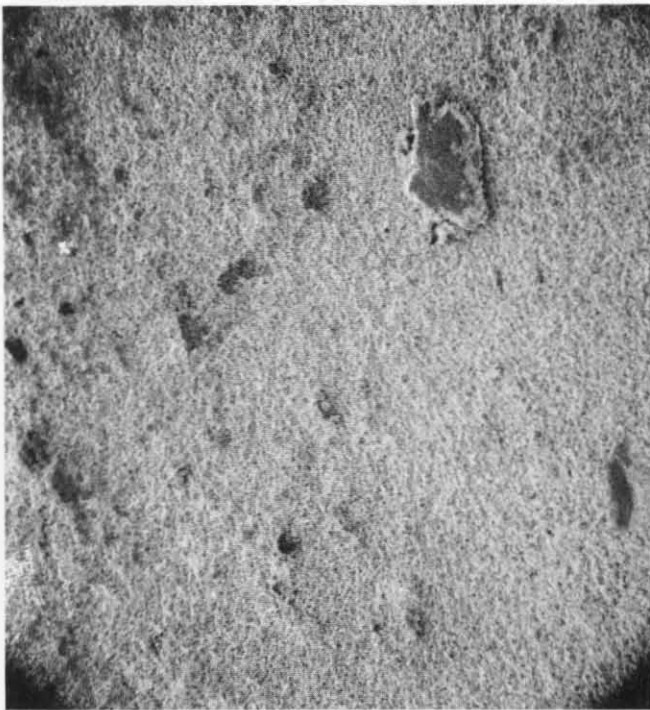


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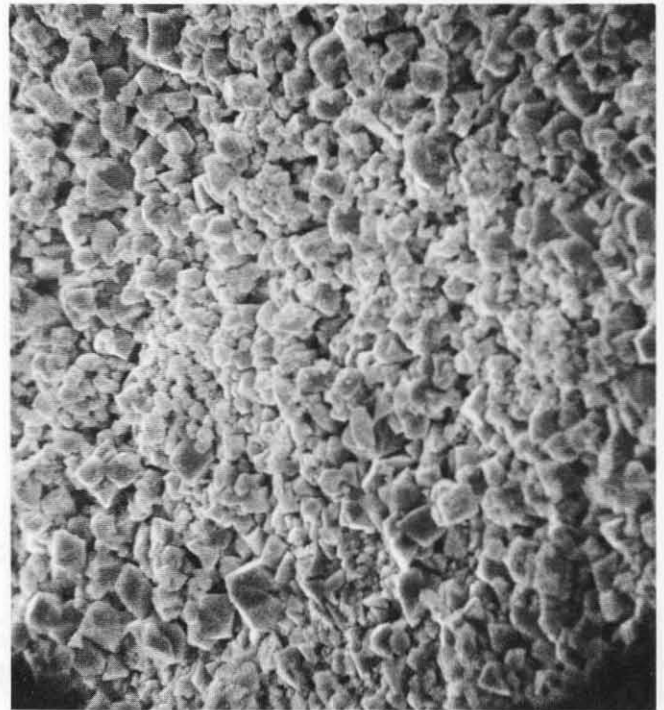


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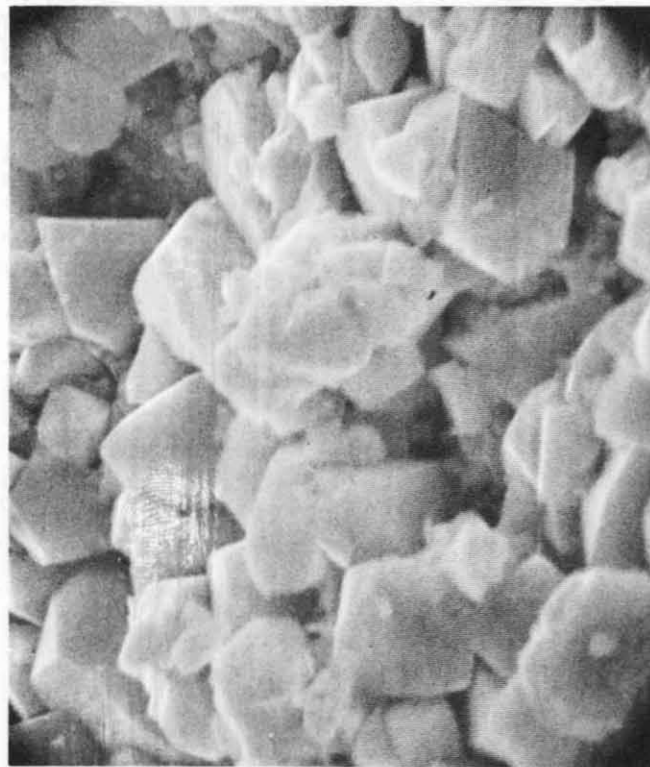
FIGURE 21. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODES MADE FROM ISOSTATICALLY COMPACTED PLAQUES 80 % 15 μ



105X



1050X



5250X

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FIGURE 22. SEM PHOTOMICROGRAPHS OF FORMED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 60 % 50 μ

TABLE 3. $\text{Cd}(\text{OH})_2$ CRYSTAL SIZE AFTER CYCLING

<u>ELECTRODE TYPE</u>	<u>SMOOTHED 50th CYCLE EFFICIENCY</u>	<u>$\bar{\sigma}$ (μ)</u>
60 % 15μ	18.7	2.3
60 % 25μ	24.1	1.4
60 % 50μ	18.8	1.0
60 % 90μ	19.0	0-1.0
80 % 15μ	34.3	9.8
80 % 25μ	37.1	6.8
80 % 50μ	34.9	7.3
80 % 90μ	33.7	4.7

It is very clear that the $\text{Cd}(\text{OH})_2$ crystal size is dependent upon the substrate porosity, the 60 % porous substrates have crystal sizes in the $0-2\mu$ range and the 80 % porous substrates have crystal sizes of about $7-10\mu$. This difference in crystal sizes is conspicuous when one compares Figures 20 and 21. It must be pointed out, however, that the crystal size is perhaps confounded with the depth of discharge and perhaps porosity of the two different groups of electrodes. One would generally expect better efficiencies for electrodes with the smaller crystal sizes, yet the opposite was observed.

No definite relationship between crystal size and electrode efficiency was apparent. A trend of decreasing crystal size with increasing induced pore size was evident.

As an additional point of interest, the formed but uncycled electrodes had average crystal sizes of 3.0μ , so that upon cycling, crystal sizes of the electrodes with the 60 % porous substrates decreased while those with the 80 % porous substrates increased.

At this point, it is worthwhile to repeat that the above treatment of crystal size is approximate because of the difficulty in interpreting the size of the crystals.

It thus appears, if we may say from such a limited quantity of data, that the $\text{Cd}(\text{OH})_2$ crystal size in these electrodes at the loading levels studied is dependent upon both the plaque pore size and porosity.

F. X-RAY RADIOGRAPHY OF PLAQUES AND ELECTRODES

The intensity of an X-ray beam is attenuated locally as it passes through a homogeneous or heterogeneous body. This is due to the adsorption or scattering of the X-ray beam by the material concerned. As a result, the X-ray beam emerging from the object can form, at the surface of a photographic film, areas of differing intensity which as a whole comprise the X-ray image. This is the so-called technique of X-ray radiography. It was used here as a qualitative tool for the measurement of microscopic uniformity of plaques prepared, and followed through after loading with active material, formation, and cycling. This was done in an effort to monitor the bulk uniformity of components in a qualitative way at various stages in the electrode testing and processing regime. Measurements were made on virgin plaque, formed or unformed virgin electrodes, and the electrode at the conclusion of the testing.

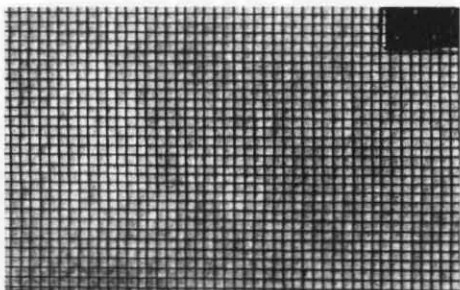
The intensity of the radiographs in the format in which they are given is dependent on numerous factors. Some of the more important factors are the intensity of the X-ray beam, exposure time (on negative and print), film type, etc. These are aside from the optical density of the specimen material. Since we were only interested in density gradations within a particular specimen, no rigorous effort was made to carefully control the photographic steps so that a given intensity on the radiograph would correspond to an actual density value. As a result, comparing densities from specimen to specimen has no meaning since these are biased by the photographic processing.

Radiographs representing samples of each porosity and pore-size are shown in Figures 24 through 32. Figure 23 shows radiographs of state-of-the-art electrodes induced for comparison. A careful visual comparison shows several interesting features. First, the bulk uniformity of the isostatically compacted plaques was good, compared to state-of-the-art. An increasing degree of uniformity was evident as the size of the pore-former was increased. This was probably due to increased ease of processing and preparing a uniform powder blend. Uniformity of the isostatically compacted plaques could still be improved by tightening of the process variables; no exceptional effort could be made to this end since so many other parameters had to be defined in this exploratory study.

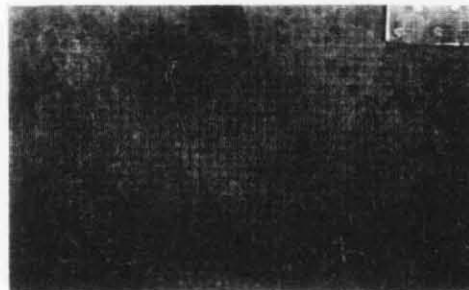
A trend toward increased uniformity with increased induced pore size was also noted with the impregnated plaques. The apparent lack of uniformity of some of the impregnated plaques is due to the impregnation procedure employed. Particularly noticeable is the mottling of the plaques, and high density areas on one edge. This high density area on one edge is due to attitude effects during impregnation. The electrodes were prepared in racks standing on edge², allowing gravitational effects to force the higher loading on the lowest edge. The mottling effect can be attributed to the process itself and apparently not to the plaques. Modified impregnation procedures designed to the need of the particular type of plaques may be required to improve uniformity of electrodes. The cycle test results were not affected by these particular nonuniformities.

Further, examination of the radiographs of these same electrodes after cycling, showed that they were identical to the radiographs of the unformed or formed electrodes. This indicated that there were no bulk material location changes in the electrode during cycle testing.

Another interesting aspect of these electrodes, which was made very apparent by the radiographs of the electrodes after cycling, was the amount of cracking observed. A large part of these cracks were apparent upon visual examination, but others were invisible subsurface cracks. Cracking during cycle test can be detrimental to the performance of the electrode due to increasing internal resistance and general reduction in current collection efficiency. The exact cause of this phenomena is not known; however, it may be due to electrode dimensional changes generating stresses during charge and discharge. Also, it is very important to note that the 80 % porous substrate material was far more susceptible to cracking (see Mechanical Strengths in Figure 5) than was the stronger 60 % porous substrate materials. The gravity sintered (state-of-the-art) exhibited the most severe cracking.



Raw Plaque Prepared By
Gravity Sintering Technique



Unformed Electrode Prepared
From A Gravity Sintered Plaque

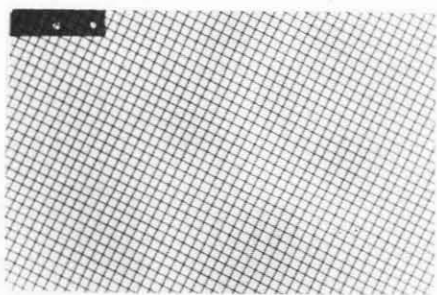


Formed, Uncycled Electrode

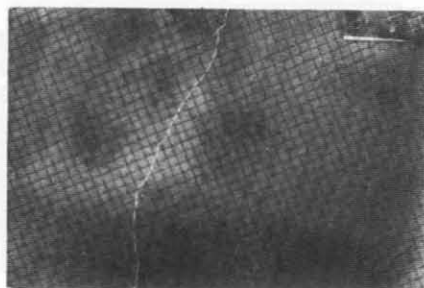


Cycled Electrode

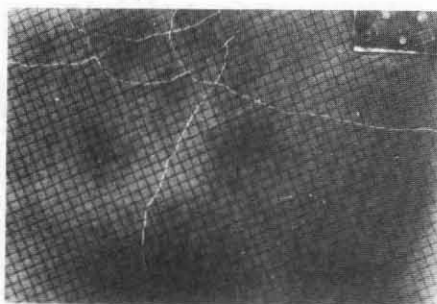
FIGURE 23. TYPICAL "STATE-OF-THE-ART" ELECTRODE
PREPARED FROM GRAVITY SINTERED PLAQUE



**Raw Plaque Prepared
By Isostatic Compaction**

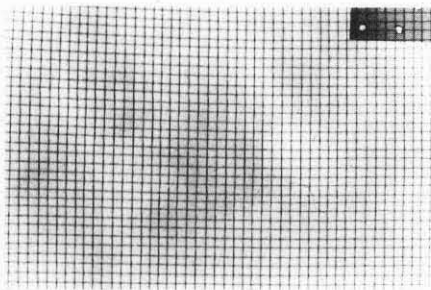


**Formed Electrode Prepared From An
Isostatically Compacted Plaque**

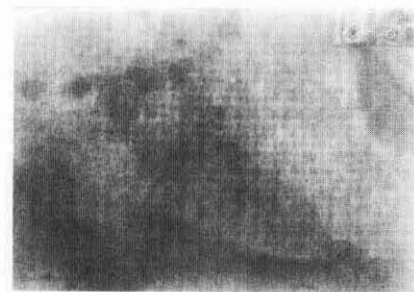


Cycled Electrode After 56 Cycles

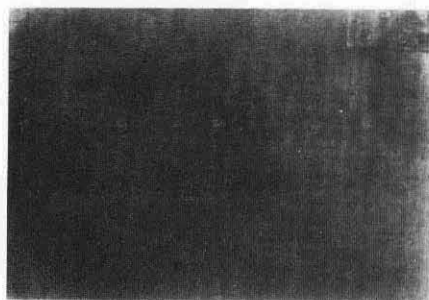
**FIGURE 24. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE, 80 % 15 μ PORE SIZE**



Raw Plaque Prepared
By Isostatic Compaction

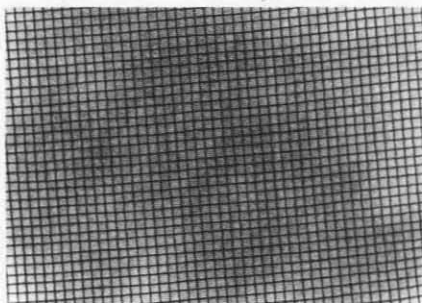


Formed Electrode Prepared From An
Isostatically Compacted Plaque

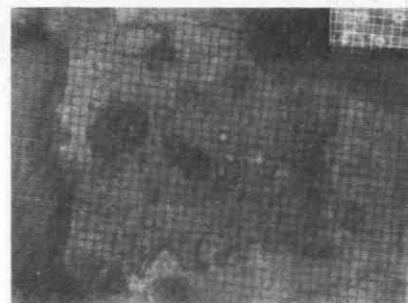


Cycled Electrode After 56 Cycles

**FIGURE 25. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE, 60 % 15 μ INDUCED PORE SIZE**



Raw Plaque Prepared
By Isostatic Compaction

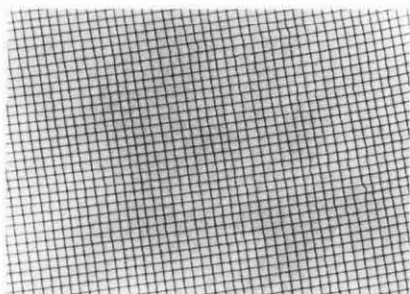


Unformed Electrode Prepared From An
Isostatically Compacted Plaque

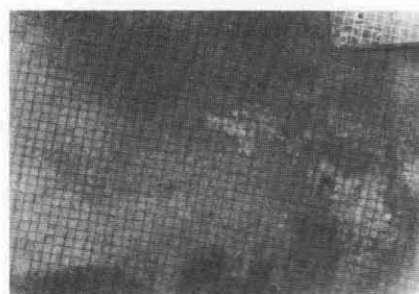


Cycled Electrode After 56 Cycles

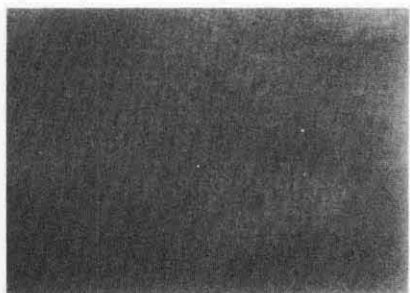
FIGURE 26. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE, 80 % 25 μ INDUCED PORE SIZE



**Raw Plaque Prepared
By Isostatic Compaction**



Unformed Electrode

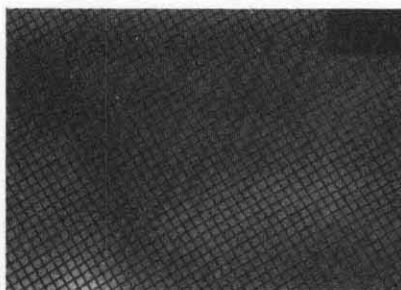


Formed Electrode

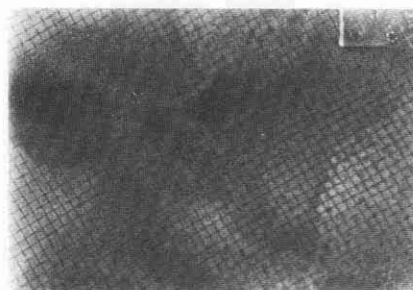


Cycled Electrode After 60 Cycles

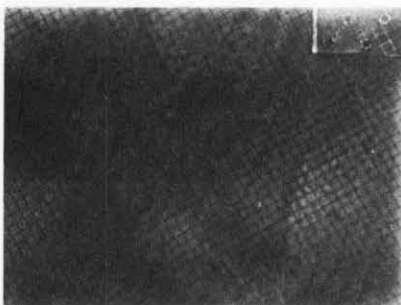
**FIGURE 27. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE 60 % 25 μ INDUCED PORE SIZE**



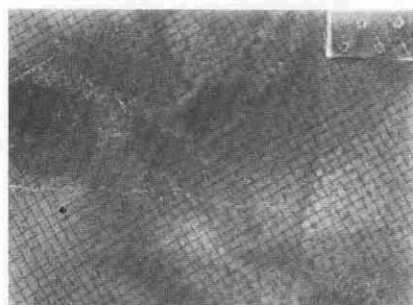
Raw Plaque Prepared
By Isostatic Compaction



Unformed Electrode

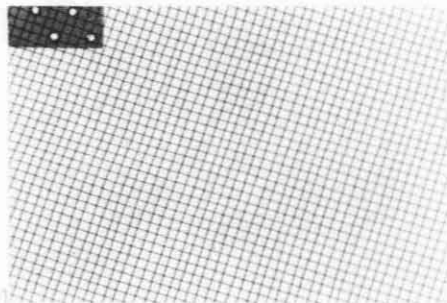


Formed Electrode



Cycled Electrode After 56 Cycles

FIGURE 28. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE 80 % 25 μ INDUCED PORE SIZE, BLENDED POWDER

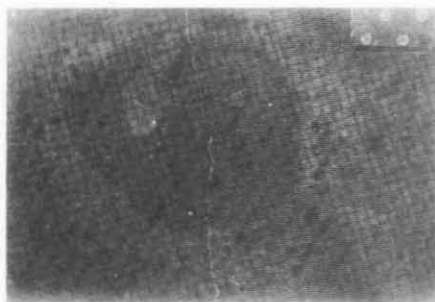


Raw Plaque Prepared
By Isostatic Compaction



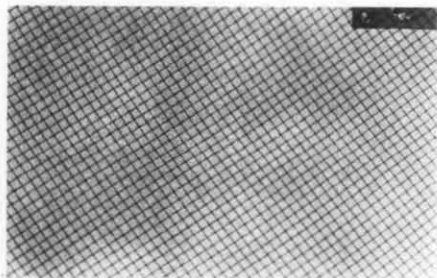
Unformed Electrode

Reproduced from
best available copy.

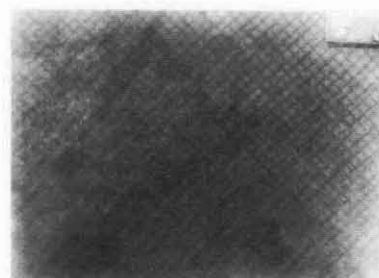


Cycled Electrode After 56 Cycles

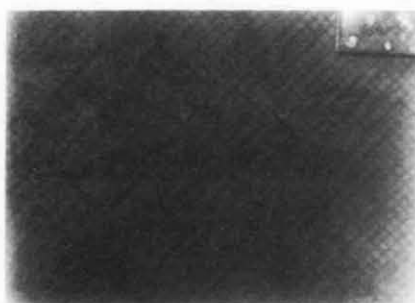
FIGURE 29. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE, 80 % 50 μ INDUCED PORE SIZE



**Raw Plaque Prepared
By Isostatic Compaction**

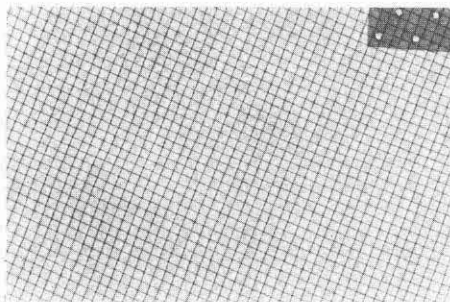


Unformed Electrode

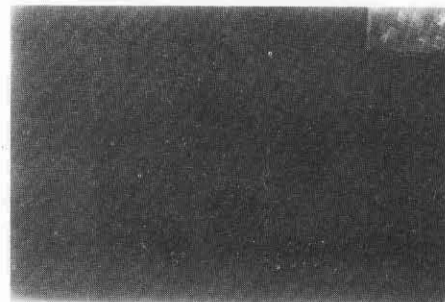


Cycled Electrode After 56 Cycles

**FIGURE 30. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE 60 % 50 μ INDUCED PORE SIZE**

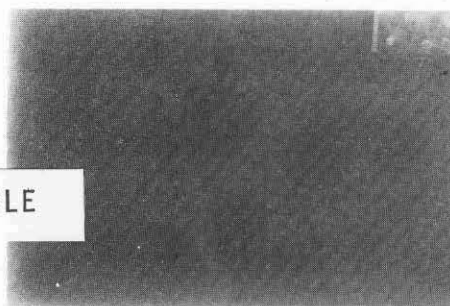


**Raw Plaque Prepared
By Isostatic Compaction**



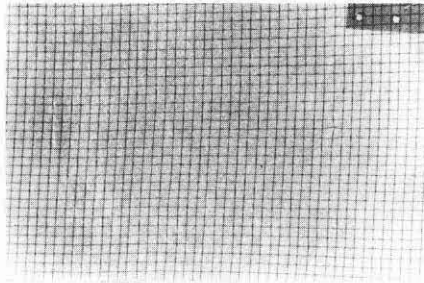
Unformed Electrode

NOT REPRODUCIBLE

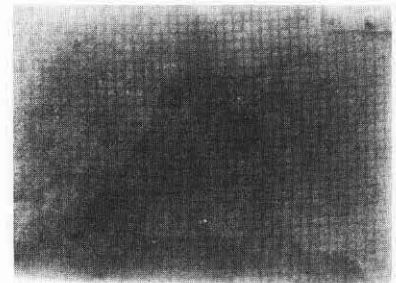


Cycled Electrode After 56 Cycles

**FIGURE 31. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE, 80 % 90 μ INDUCED PORE SIZE**

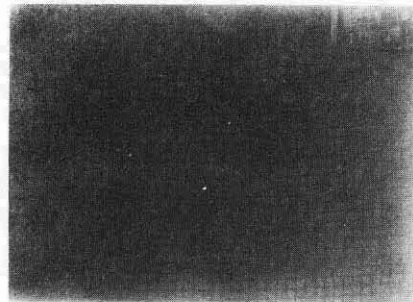


**Raw Plaque Prepared
By Isostatic Compaction**



Unformed Electrode

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best available copy.



Cycled Electrode After 56 Cycles

**FIGURE 32. ELECTRODE PREPARED FROM ISOSTATICALLY
COMPACTED PLAQUE 60 % 90 μ INDUCED PORE SIZE**

IV. CONCLUSIONS

An exploratory study of a technique for preparing improved cadmium electrode substrates with precisely controlled microstructures for possible use in aerospace nickel-cadmium cells has been completed. Cadmium electrodes were prepared having 60 % and 80 % porosities, each at 15, 25, and 90 μ induced pore size levels, and cycle tested using an accelerated regime. The results reveal several very good prospects for improved aerospace cadmium electrodes. These are:

1. Cadmium electrodes prepared from 60 % porosity 25 μ induced pore size plaques had efficiencies only 5-10 percentage points lower than state-of-the-art electrodes judging from the results of an accelerated test regime. These plaques had vastly superior mechanical properties, about 8000 psi as compared to 645 psi for the conventional gravity sintered material in use today. This added strength was observed to eliminate cracking and physical degradation of the electrodes during processing and cycling. The electrical performance of these electrodes was poorer than state-of-the-art electrodes.

It is reasonable to assume that efficiencies of such electrodes may be raised without greatly sacrificing mechanical properties.

2. Cadmium electrodes prepared from 80 % porous 25 μ induced pore size plaques, under the tortuous test regime employed, proved to be the best electrodes made during the course of this program from the point of view of highest efficiency. They were superior to state-of-the-art electrodes in this respect, in addition to having somewhat superior mechanical strengths and better uniformity.
3. Of a more developmental type of interest that may be refined to yield practical results, it was observed that:
 - The plaque pore size and porosity were found to be contributing factors to $\text{Cd}(\text{OH})_2$ crystal size, reportedly an important property governing the performance and life of cadmium electrodes. No relation between crystal size and efficiency was found.
 - The induced porosity was observed to be a factor in electrode performance. Higher induced porosity apparently yielded better electrodes. This property was confounded with loading, however.
 - The best performance at both the 60 % and 80 % porosities were observed with the 25 μ induced sizes. The higher induced pore sizes, the 50 and 90 μ , were more uniform as shown in X-ray radiographs.
 - Morphological changes of $\text{Cd}(\text{OH})_2$, determined from SEM photomicrographs, were identified after the cycling procedure. With the 60 % structures the crystals apparently decreased in size with cycling, while with the 80 % porosity structures the crystals increased in size.

- Active material loading uniformity on the bulk scale was measured in the X-ray radiographs. There were no observable bulk changes in uniformity with cycling.
- Some small degree of corrosion of the plaques was evident from photomicrographs. The corrosion was due either to processing or cycling.
- Plaques were thoroughly characterized with the use of the SEM. Induced pore-sizes were evaluated, interstitial pores described and various fine structures, pits and scars, identified.
- BET surface areas were measured and varied between 0.11 and 0.17 m²/gm depending upon plaque pore size and porosity.
- Three-point bend tests were performed on selected specimens and varied between about 900 and 8000 psi between porosities of 80 % and 60 %. As a point of comparison, a 79 % gravity sintered material had a bend strength of 643 psi.

V. RECOMMENDATIONS FOR FUTURE WORK

An initial study of improved plaque materials prepared via a powder metallurgical technique allowing control over porosity and pore size has yielded cadmium electrodes that are better than state-of-the-art electrodes, in terms of efficiency and mechanical strength. There are a number of areas that are worthy of exploration that are likely to yield still further improved cadmium electrodes with respect to reproducibility, uniformity, and long life.

1. Optimize performance of high strength substrates; e.g., 60 % porous, to the level of the 80 % structures. This should involve improved processing steps. Increased induced porosities should help also.
2. Better active material loading technique to attain more uniform, more efficient active material loading.
3. Evaluate selected optimized substrate more thoroughly; i.e., much longer cycle life testing in practical cells over a wider range of test conditions.
4. Determine effect of pore size on oxygen recombinations in practical cells.
5. Further develop techniques for mold filling and powder handling and classification techniques to improve reproducibility and uniformity of the plaque.

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APPENDIX A
SUMMARY OF MECHANICAL STRENGTH DATA

Table 4

Results of a Least Squares Fit on
Mechanical Strength Data

Actual Powder Porosity	Measured Mechanical Strength	Calculated Mechanical Strength	Percent Difference	Induced Pore-Size
56.77 %	9014 psi	8197 psi	- 9.0	25 μ
58.21	8803	7802	-11.3	15 μ
58.46	8225	7734	- 5.9	15 μ
59.68	7747	7401	- 4.4	15 μ
63.68	5368	6310	+17.5	90 μ
64.00	6267	6222	- 0.7	50 μ
64.94	5488	5965	+ 8.7	90 μ
65.01	5995	5946	- 0.8	25 μ
65.55	5080	5799	+14.1	25 μ
66.26	5578	5606	+ 0.4	25 μ
66.34	5636	5584	- 0.9	90 μ
66.53	6080	5531	- 9.0	50 μ
67.23	4257	5340	+25.4	15 μ
67.42	4763	5289	+11.0	15 μ
68.46	4375	5002	+14.3	15 μ
81.88	1265	1340	+ 5.9	90 μ
82.50	917	1172	+27.7	25 μ
82.64	1100	1134	+ 3.0	25 μ
82.82	946	1085	+14.7	25 μ
82.86	1048	1075	+ 2.5	25 μ
82.89	841	1066	+26.7	25 μ
83.96	1044	773	-25.9	90 μ
84.00	828	763	- 7.8	90 μ
84.77	788	551	-30.1	50 μ
84.93	750	508	-32.2	25 μ
84.97	800	498	-37.7	50 μ
84.98	550	495	- 9.9	50 μ
85.11	776	459	-40.7	25 μ
85.11	411	459	+11.6	25 μ
85.41	747	377	-49.5	15 μ

v

APPENDIX B
SUMMARY OF CYCLE TEST DATA FOR
"STATE-OF-THE-ART" ELECTRODES

TABLE 5

SUMMARY OF CYCLE TEST DATA FOR "STATE-OF-THE-ART" ELECTRODES

"STATE OF THE ART"
Gould Standard Process

"STATE OF THE ART"
Precipitation Process

Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency	Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	1.1218	56.688	±2.082	1	1.1124	60.638	±5.462
2	1.1406	57.638	±2.208	2	1.1006	59.975	±5.425
3	1.1613	58.675	±2.771	3	1.0301	56.138	±5.545
4	0.8616	43.525	±3.519	4	0.7269	39.513	±9.339
5	0.6610	33.375	±4.405	5	0.5184	28.150	±10.432
6	0.3035	15.263	±5.030	6	0.2634	14.357	±5.196
7	0.7494	37.851	±3.187	7	0.6222	38.300	±3.141
8	0.7733	39.046	±3.442	8	0.6388	38.579	±2.952
9	0.7629	38.514	±3.608	9	0.6386	38.804	±2.908
10	0.7397	37.344	±3.617	10	0.6276	38.257	±3.307
11	0.7547	38.109	±3.023	11	0.6201	38.170	±3.439
12	0.7434	37.538	±3.315	12	0.6147	37.678	±3.765
13	0.7235	36.529	±3.293	13	0.6060	37.278	±3.869
14	0.7153	36.109	±3.467	14	0.5951	36.883	±4.034
15	0.7046	35.567	±3.445	15	0.5972	36.816	±4.202
16	0.6932	35.000	±3.288	16	0.5832	36.032	±4.236
17	0.6780	34.219	±3.504	17	0.5675	35.691	±4.373
18	0.6646	33.546	±3.452	18	0.5519	35.142	±4.114
19	0.6466	32.635	±3.183	19	0.6044	37.228	±4.149
20	0.7093	35.841	±2.555	20	0.5985	36.787	±4.255
21	0.6967	35.203	±2.365	21	0.6422	39.757	±4.196
22	0.6974	35.239	±1.952	22	0.5951	36.692	±4.157
23	0.6896	34.829	±1.957	23	0.6479	39.057	±4.097
24	0.7039	35.558	±1.902	24	0.5963	36.609	±4.534
25	0.6966	35.189	±2.291	25	0.5799	35.760	±4.352
26	0.6941	35.063	±2.357	26	0.5560	34.715	±4.343
27	0.6818	34.437	±2.234	27	0.5456	34.016	±4.458
28	0.6714	33.915	±2.245	28	0.5262	32.997	±4.420
29	0.6650	33.585	±2.255	29	0.5310	33.627	±4.633
30	0.6535	32.999	±2.411	30	0.5209	33.019	±4.670
31	0.6381	32.222	±2.380	31	0.5162	32.727	±3.735
32	0.6255	31.589	±2.403	32	0.5026	31.755	±4.653
33	0.6161	31.111	±2.545	33	0.5476	34.607	±3.984
34	0.6058	30.589	±2.505	34	0.5583	34.929	±4.129
35	0.6784	34.278	±2.015	35	0.5640	35.092	±4.028
36	0.6714	33.924	±1.926	36	0.5586	34.683	±4.041
37	0.6781	34.271	±1.814	37	0.5783	36.346	±5.560
38	0.6839	34.567	±1.497	38	0.5605	34.808	±4.080
39	0.6910	34.928	±1.106	39	0.5511	34.363	±4.087
40	0.6801	34.365	±1.416	40	0.5441	33.910	±4.134
41	0.6678	33.742	±1.731	41	0.5334	33.401	±4.076
42	0.6567	33.180	±1.434	42	0.5256	32.937	±4.298
43	0.6439	32.534	±1.729	43	0.5165	32.436	±4.401
44	0.6333	31.994	±1.834	44	0.5004	31.870	±4.387
45	0.6321	31.930	±1.915	45	0.4936	31.304	±4.383
46	0.6170	31.170	±1.730	46	0.4854	31.045	±4.438
47	0.6089	30.759	±1.839	47		Recorder Failure	
48	0.6404	32.368	±2.044	48	0.4567	29.430	±4.642
49	0.6348	32.086	±2.128	49	0.4424	28.724	±4.574
50	0.6609	33.428	±1.505	50	0.4265	28.141	±4.786
51	0.6594	33.351	±1.590	51	0.4221	27.786	±5.018
52	0.6641	33.591	±1.541	52	0.4090	27.131	±5.072
53	0.6669	33.731	±1.448	53	0.3911	26.135	±5.138
54	0.6658	33.679	±1.477	54	0.3856	25.817	±5.338
55	0.6597	33.375	±1.396	55	0.3731	25.085	±5.802
56	0.6522	32.994	±1.494	56	0.3636	24.650	±5.889

APPENDIX C

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

TABLE 6

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

60% Porosity 15 μ Induced Pore-Size				80% Porosity 15 μ Induced Pore-Size			
Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency	Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	0.7404	39.625	± 2.523	1	0.8466	57.825	± 2.814
2	0.7501	40.113	± 2.088	2	0.7648	52.150	± 2.654
3	0.7696	41.100	± 2.414	3	0.8265	56.350	± 2.795
4	0.4844	25.850	± 2.677	4	0.6268	42.713	± 2.260
5	0.3211	17.125	± 3.528	5	0.4635	31.575	± 2.113
6	0.1373	7.313	± 3.734	6	0.3478	23.663	± 3.101
7	0.4053	21.592	± 2.168	7	0.5416	36.922	± 1.617
8	0.4102	21.849	± 2.188	8	0.5523	37.640	± 1.906
9	0.3762	21.901	± 2.282	9	0.5634	38.411	± 1.743
10	0.4009	21.338	± 2.482	10	0.5704	38.875	± 2.040
11	0.4270	22.754	± 2.235	11	0.5666	38.611	± 2.201
12	0.4182	22.284	± 2.367	12	Recorder Failure		
13	0.4064	21.648	± 2.435	13		"	
14	0.3996	21.278	± 2.398	14		"	
15	0.3915	20.834	± 2.544	15		"	
16	0.3801	20.216	± 2.715	16		"	
17	0.3730	19.836	± 2.677	17		"	
18	0.3621	19.253	± 2.812	18	0.5659	38.557	± 2.571
19	0.3062	18.226	± 2.662	19	0.5602	38.171	± 2.602
20	0.3343	17.773	± 2.715	20	0.5583	38.036	± 2.692
21	0.3324	17.728	± 3.117	21	0.5573	37.920	± 2.779
22	0.4709	22.702	± 2.330	22	0.5584	38.047	± 2.896
23	0.4209	22.432	± 2.248	23	0.5634	38.382	± 2.896
24	0.4192	22.347	± 2.285	24	0.5587	38.068	± 2.805
25	0.4105	21.860	± 2.192	25	0.5581	38.019	± 2.915
26	0.4017	21.389	± 2.347	26	0.5545	37.776	± 3.036
27	0.4151	22.130	± 2.202	27	0.5444	37.082	± 3.166
28	0.4054	21.605	± 2.028	28	0.5418	36.904	± 3.295
29	0.4065	21.656	± 2.206	29	0.5391	36.709	± 3.509
30	0.3936	20.964	± 2.078	30	0.5249	35.736	± 3.619
31	0.3897	20.743	± 2.136	31	0.5263	35.836	± 3.735
32	0.3665	19.505	± 2.317	32	0.5505	37.481	± 3.381
33	0.3573	19.010	± 2.243	33	0.5516	37.561	± 3.105
34	0.3495	18.583	± 2.337	34	0.5654	38.512	± 3.231
35	0.3320	17.649	± 2.370	35	0.5598	38.140	± 2.914
36	0.3251	17.294	± 2.149	36	0.5568	37.931	± 3.024
37	0.3045	16.191	± 2.155	37	0.5551	37.819	± 2.840
38	0.3964	21.123	± 2.009	38	0.5532	37.687	± 3.002
39	0.3947	21.032	± 1.873	39	0.5446	37.101	± 3.166
40	0.3955	21.058	± 2.181	40	0.5418	36.907	± 3.204
41	0.4037	21.509	± 1.954	41	0.5361	36.510	± 3.325
42	0.4051	21.590	± 1.975	42	0.5304	36.117	± 3.498
43	0.3959	21.098	± 1.989	43	0.5246	35.725	± 3.767
44	0.3887	20.708	± 1.945	44	0.5113	34.819	± 3.728
45	0.3839	20.453	± 2.035	45	0.5064	34.470	± 3.901
46	0.3650	19.430	± 1.859	46	0.5024	34.198	± 3.901
47	0.3583	19.074	± 1.928	47	0.4833	32.908	± 4.024
48	0.3389	18.031	± 1.983	48	0.4693	31.949	± 4.065
49	0.3374	17.950	± 2.040	49	0.4610	31.386	± 3.992
50	0.3219	17.119	± 2.056	50	0.4491	30.580	± 4.010
51	0.3038	16.181	± 2.136	51	0.4110	27.955	± 4.674
52	0.2964	15.758	± 1.956	52	0.4332	29.476	± 4.376
53	0.2892	15.359	± 2.074	53	0.4242	28.871	± 4.476
54	0.3720	19.808	± 1.964	54	0.5183	35.284	± 3.513
55	0.3786	20.174	± 1.952	55	0.5205	35.439	± 3.498
56	0.3704	19.718	± 1.991	56	0.5121	34.872	± 3.599

TABLE 7

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

60% POROSITY 25 μ INDUCED PORE SIZE				80% POROSITY 25 μ INDUCED PORE SIZE			
Cycle No.	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency	Cycle No.	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	0.7574	38.813	± 2.552	1	0.9501	54.238	± 2.529
2	0.7924	40.988	± 2.253	2	1.1013	56.463	± 2.532
3	0.8191	42.313	± 1.983	3	1.0615	38.988	± 5.175
4	0.5833	30.757	± 1.631	4	0.7640	27.488	± 6.445
5	0.3904	20.425	± 3.984	5	0.5445	16.600	± 5.203
6	0.1528	8.138	± 3.336	6	0.3271	8.138	± 3.336
7	0.5528	28.472	± 2.038	7	0.7388	41.073	± 4.874
8	0.4866	24.662	± 1.781	8	0.6943	38.546	± 10.264
9	0.6232	32.278	± 2.185	9	0.7414	41.495	± 4.954
10	0.6252	32.239	± 2.033	10	0.7380	41.369	± 4.605
11	0.6352	32.469	± 1.965	11	0.7301	40.996	± 4.867
12	0.6118	31.549	± 2.066	12	0.7257	40.729	± 4.996
13	0.6110	31.501	± 2.132	13	0.7145	40.126	± 4.990
14	0.6046	31.450	± 2.139	14	0.7062	39.800	± 5.216
15	0.6033	31.093	± 2.209	15	0.7290	40.484	± 5.242
16	0.6004	30.942	± 2.293	16	0.7302	40.830	± 4.030
17	0.5980	30.819	± 2.267	17	0.7219	40.254	± 4.002
18	0.5913	30.464	± 2.392	18	0.7257	40.269	± 3.599
19	0.5681	30.198	± 2.617	19	0.7260	40.517	± 3.402
20	0.5877	30.330	± 2.109	20	0.7141	40.007	± 3.486
21	0.5775	29.801	± 2.063	21	0.7075	39.684	± 3.275
22	0.5726	29.812	± 2.141	22	0.7025	39.297	± 3.332
23	0.5639	29.156	± 2.190	23	0.6902	38.830	± 3.480
24	0.5662	29.250	± 2.121	24	0.6811	38.376	± 3.597
25	0.5545	28.640	± 2.139	25	0.6746	38.119	± 3.675
26	0.5625	28.982	± 1.931	26	0.7018	38.829	± 4.109
27	0.5421	28.370	± 2.049	27	0.7113	39.549	± 3.060
28	0.5591	28.816	± 1.990	28	0.7185	39.735	± 2.714
29	0.5493	28.329	± 2.131	29	0.7127	39.579	± 2.729
30	0.5432	28.195	± 2.094	30	0.7146	39.468	± 2.894
31	0.5464	28.177	± 2.193	31	0.7096	39.382	± 2.734
32	0.5320	27.426	± 2.206	32	0.7002	39.038	± 2.739
33	0.5266	27.160	± 2.229	33	0.6964	38.821	± 2.815
34	0.5294	27.381	± 2.183	34	0.6915	38.583	± 3.055
35	0.5121	26.758	± 2.511	35	0.6788	37.953	± 3.054
36		Recorder Failure		36	0.6641	37.163	± 3.091
37	0.4232	26.706	± 2.287	37	0.6503	36.481	± 3.166
38		Recorder Failure		38	0.7017	38.761	± 2.613
39		Recorder Failure		39	0.6907	38.520	± 2.872
40	0.5060	26.126	± 2.080	40	0.7025	38.789	± 3.044
41	0.4956	25.610	± 2.019	41	0.6982	28.687	± 3.115
42	0.4997	25.815	± 1.910	42	0.7031	38.754	± 3.063
43	0.4766	25.375	± 2.089	43	0.6903	38.256	± 2.899
44	0.4793	25.070	± 2.014	44	0.6823	37.933	± 3.031
45	0.4900	25.286	± 1.806	45	0.6759	37.492	± 3.100
46	0.4851	24.646	± 1.694	46	0.6617	36.798	± 3.327
47	0.4763	24.887	± 2.331	47	0.6565	36.592	± 3.384
48	0.4718	24.311	± 2.326	48	0.6408	35.747	± 3.561
49	0.4660	24.028	± 2.156	49	0.6402	35.745	± 3.676
50	0.4586	23.638	± 2.469	50	0.6827	37.661	± 3.194
51	0.4504	23.307	± 2.634	51	0.6780	37.544	± 3.044
52	0.4498	23.177	± 2.725	52	0.6896	37.940	± 3.400
53	0.4308	22.190	± 2.800	53	0.6745	37.274	± 3.242
54	0.4322	22.196	± 2.826	54	0.6776	37.229	± 3.183
55	0.4530	23.424	± 2.291	55	0.6684	36.961	± 3.133
56	0.4507	23.280	± 2.136	56	0.6518	36.264	± 2.901

TABLE 8

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

80% Porosity 25 μ Induced Pore-Size (Blended Powders)			
Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	Recorder Failure		
2	0.9163	55.721	± 1.903
3	0.9174	55.750	± 1.846
4	0.7130	43.338	± 2.201
5	0.5336	32.375	± 3.249
6	0.2764	16.825	± 4.838
7	0.6522	40.078	± 3.731
8	0.6716	41.275	± 3.864
9	0.6627	41.308	± 4.527
10	0.6607	41.324	± 4.288
11	0.6472	40.719	± 4.540
12	0.6466	40.304	± 4.458
13	0.6422	40.030	± 4.387
14	0.6207	39.761	± 4.325
15	0.6352	39.601	± 4.225
16	0.6270	39.092	± 4.088
17	0.6255	39.000	± 3.982
18	0.6031	37.932	± 4.551
19	0.6295	38.868	± 4.067
20	0.6373	39.146	± 3.652
21	0.6630	39.203	± 3.614
22	0.6312	38.773	± 3.576
23	0.6329	38.883	± 3.454
24	0.6293	38.667	± 3.418
25	0.6230	38.287	± 3.249
26	0.6152	37.801	± 3.371
27	0.6196	38.078	± 3.218
28	0.6125	37.643	± 3.192
29	0.6052	37.197	± 3.037
30	0.6048	37.184	± 2.918
31	0.5987	36.802	± 2.946
32	0.5957	36.627	± 2.835
33	0.5919	36.396	± 2.767
34	0.5857	36.022	± 2.719
35	0.5896	36.262	± 2.633
36	0.5816	35.769	± 2.620
37	0.5836	35.895	± 2.525
38	0.5827	35.845	± 2.473
39	0.5844	35.957	± 2.385
40	0.5802	35.698	± 2.419
41	0.5775	35.536	± 2.336
42	0.5763	35.467	± 2.198
43	0.5706	35.123	± 2.105
44	0.5719	35.201	± 2.145
45	0.5701	35.084	± 2.181
46	0.5727	35.253	± 2.115
47	0.5723	35.229	± 2.159
48	0.5693	35.046	± 2.074
49	0.5661	34.850	± 2.045
50	0.5643	34.739	± 2.043
51	0.5614	34.557	± 2.100
52	0.5638	34.713	± 1.997
53	0.5688	35.015	± 2.065
54	0.5715	35.183	± 2.074
55	0.5670	34.905	± 2.079
56	0.5658	34.836	± 2.053

TABLE 9

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

60% Porosity 50 μ Induced Pore-Size				80% Porosity 50 μ Induced Pore-Size			
Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency	Cycle Number	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	0.7916	40.636	± 2.037	1	0.9870	45.572	± 2.269
2	0.7970	40.928	± 1.325	2	0.9556	44.146	± 3.194
3	0.7922	40.688	± 1.143	3	0.9012	41.600	± 2.493
4	0.3908	19.924	± 5.091	4	0.7556	34.886	± 1.386
5	0.1878	9.538	± 4.455	5	0.5606	25.876	± 1.992
6	0.0886	4.500	± 2.427	6	0.0466	2.156	± 0.533
7	0.3886	19.869	± 3.883	7	0.6486	29.944	± 2.094
8	0.3891	19.894	± 3.563	8	0.6788	31.341	± 2.016
9	0.4130	21.138	± 3.570	9	0.6794	31.374	± 1.569
10	0.4161	21.281	± 3.724	10	0.6897	31.849	± 1.483
11	0.4022	20.562	± 4.374	11	0.6968	32.181	± 1.461
12	0.3904	19.954	± 4.845	12	0.7006	32.355	± 1.497
13	0.4276	21.861	± 3.956	13	0.7115	32.858	± 1.547
14	0.4075	20.843	± 3.719	14	0.7249	33.472	± 2.057
15	0.4216	21.569	± 3.624	15	0.7295	33.682	± 2.197
16	0.3930	20.089	± 4.988	16	0.7334	33.862	± 2.246
17	0.4335	22.188	± 4.232	17	0.7365	34.002	± 2.573
18	0.4012	20.519	± 3.928	18	0.7465	34.472	± 2.123
19	0.3949	20.179	± 5.210	19	0.7357	33.968	± 2.212
20	0.4207	21.526	± 3.603	20	0.7353	33.950	± 2.148
21	0.3989	20.393	± 4.803	21	0.7399	34.163	± 2.239
22	0.4170	21.338	± 3.473	22	0.7407	34.201	± 2.163
23	0.4165	21.295	± 3.556	23	0.7439	34.350	± 2.164
24	0.4075	20.836	± 3.723	24	0.7400	34.168	± 2.278
25	0.3861	19.733	± 5.258	25	0.7503	34.645	± 2.128
26	0.4100	22.974	± 4.788	26	0.7478	34.532	± 2.066
27	0.4062	20.772	± 3.417	27	0.7523	34.738	± 2.106
28	0.4007	20.487	± 3.629	28	0.7531	34.778	± 2.058
29	0.4186	21.421	± 3.213	29	0.7510	34.683	± 1.932
30	0.4015	20.531	± 3.344	30	0.7521	34.732	± 1.971
31	0.4018	20.554	± 3.219	31	0.7538	34.815	± 1.910
32	0.3986	20.385	± 3.295	32	0.7462	34.461	± 1.978
33	0.3994	20.438	± 3.195	33	0.7480	34.550	± 1.675
34	0.3981	20.358	± 3.339	34	0.7507	34.670	± 2.091
35	0.3820	19.529	± 3.719	35	0.7461	34.474	± 1.917
36	0.3820	19.528	± 3.714	36	0.7556	34.897	± 1.966
37	0.3778	19.328	± 3.398	37	0.7542	34.836	± 1.878
38	0.3876	19.836	± 2.741	38	0.7526	34.759	± 2.022
39	0.3823	19.566	± 2.769	39	0.7488	34.592	± 1.830
40	0.3718	19.016	± 2.972	40	0.7491	34.603	± 1.749
41	0.3826	19.578	± 2.586	41	0.7469	34.508	± 1.633
42	0.3771	19.308	± 2.399	42	0.7494	34.623	± 1.573
43	0.3693	18.895	± 2.647	43	0.7439	34.369	± 1.774
44	0.3647	18.663	± 2.759	44	0.7472	34.526	± 1.688
45	0.3684	18.855	± 2.357	45	0.7461	34.472	± 1.810
46	0.3571	18.273	± 2.640	46	0.7430	34.331	± 1.784
47	0.3502	17.907	± 3.546	47	0.7458	34.455	± 1.884
48	0.3693	18.895	± 2.706	48	0.7449	34.415	± 1.898
49	0.3627	18.563	± 2.454	49	0.7458	34.456	± 1.940
50	0.3668	18.785	± 2.305	50	0.7475	34.537	± 1.869
51	0.3599	18.416	± 2.592	51	0.7452	34.428	± 1.994
52	0.3668	18.777	± 2.617	52	0.7432	34.337	± 2.093
53	0.3607	18.459	± 2.472	53	0.7446	34.405	± 2.024
54	0.3599	18.419	± 2.479	54	0.7470	34.415	± 2.098
55	0.3385	17.312	± 3.581	55	0.7500	34.652	± 2.016
56	0.3660	18.731	± 2.648	56	0.7465	34.489	± 2.046

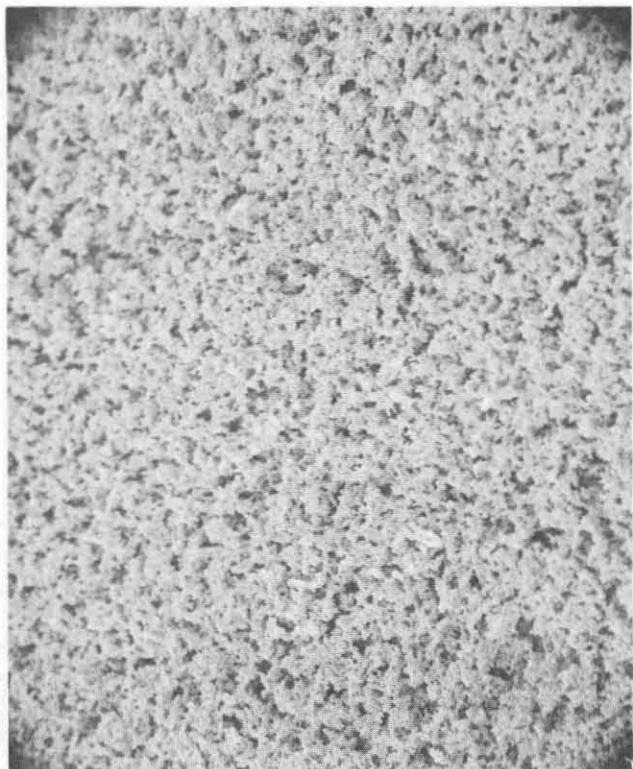
TABLE 10

SUMMARY OF CYCLE TEST DATA FOR ELECTRODES PREPARED FROM ISOSTATICALLY COMPACTED PLAQUES

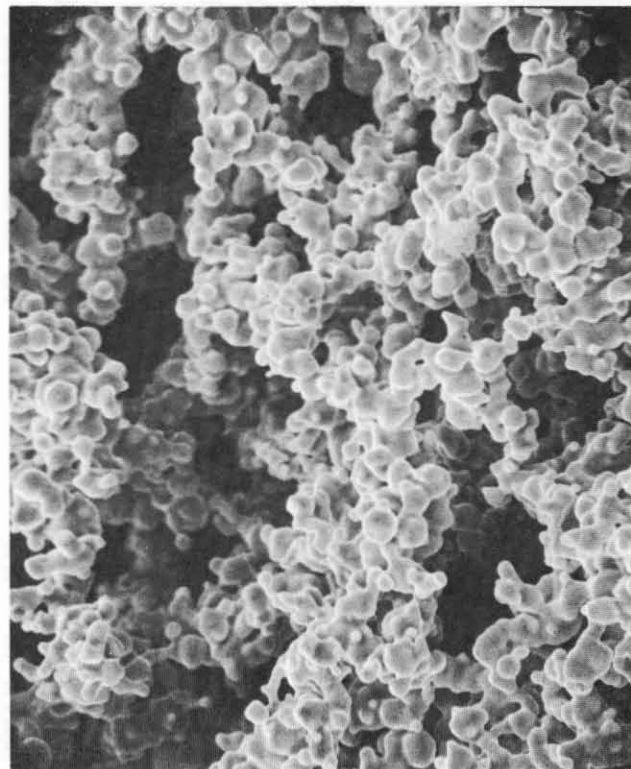
60% POROSITY 90μ INDUCED PORE SIZE				80% POROSITY 90μ INDUCED PORE SIZE			
Cycle No.	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency	Cycle No.	Average Capacity	Average Efficiency	95% Confidence Limit for Efficiency
1	0.7600	38.026	±2.697	1	1.4996	54.284	±3.331
2	0.7784	38.928	±2.293	2	1.4960	54.184	±2.066
3	0.7744	38.772	±2.282	3	1.4438	52.318	±1.556
4	0.4448	22.318	±4.599	4	1.1030	40.006	±1.166
5	0.2526	12.698	±4.569	5	0.7840	28.410	±2.074
6	0.1072	5.410	±2.751	6	0.0912	3.324	±2.338
7	0.4210	21.133	±3.974	7	0.9544	34.602	±1.292
8	0.4418	22.233	±5.034	8	1.0088	36.574	±0.939
9	0.4398	22.077	±3.946	9	1.0136	36.738	±0.897
10	0.4383	22.006	±4.001	10	1.0242	37.134	±0.434
11	0.4454	22.364	±4.139	11	1.0266	37.383	±0.495
12	0.4442	22.293	±3.865	12	1.0278	37.271	±0.632
13	0.4410	22.132	±3.942	13	1.0329	37.464	±0.886
14	0.4400	22.089	±3.889	14	1.0343	37.525	±0.636
15	0.4412	22.138	±3.747	15	1.0308	37.386	±0.473
16	0.4363	21.905	±3.854	16	1.0328	37.462	±0.563
17	0.4399	22.076	±3.619	17	1.0233	37.129	±0.823
18	0.4356	21.856	±3.690	18	1.0201	37.016	±0.807
19	0.4380	21.977	±3.605	19	1.0028	36.388	±0.822
20	0.4364	21.894	±3.622	20	0.9947	36.087	±0.663
21	0.4361	21.872	±3.341	21	0.9995	36.263	±0.676
22	0.4283	21.495	±3.658	22	0.9921	35.997	±0.766
23	0.4334	21.738	±3.478	23	0.9957	36.119	±0.594
24	0.4285	21.490	±3.368	24	0.9844	35.716	±0.786
25	0.4250	21.316	±3.379	25	0.9931	36.022	±0.678
26	0.4213	21.127	±3.291	26	0.9926	35.997	±0.582
27	0.4212	21.119	±3.290	27	0.9933	36.029	±0.634
28	0.4199	21.048	±3.120	28	0.9873	35.810	±0.838
29	0.4226	21.188	±3.168	29	0.9848	35.723	±0.693
30	0.4115	20.629	±3.155	30	0.9887	35.858	±0.788
31	0.4079	20.448	±3.129	31	0.9852	35.727	±0.767
32	0.4069	20.399	±3.082	32	0.9625	34.927	±1.584
33	0.4102	20.557	±2.933	33	0.9656	35.023	±1.436
34	0.4080	20.451	±3.020	34	0.9604	34.835	±1.684
35	0.4019	20.143	±3.045	35	0.9529	34.562	±1.844
36	0.4022	20.157	±3.055	36	0.9592	34.785	±1.918
37	0.3999	20.032	±2.748	37	0.9579	34.735	±1.949
38	0.3994	20.006	±2.947	38	0.9569	34.702	±2.049
39	0.3966	19.867	±2.888	39	0.9545	34.610	±1.924
40	0.3956	19.819	±2.903	40	0.9574	34.716	±1.946
41	0.3949	19.780	±2.759	41	0.9438	34.227	±2.065
42	0.3940	19.734	±2.802	42	0.9439	34.228	±1.994
43	0.3881	19.430	±2.598	43	0.9380	34.009	±1.980
44	0.3872	19.393	±2.647	44	0.9404	34.095	±2.091
45	0.3855	19.305	±2.711	45	0.9391	34.047	±2.051
46	0.3774	18.896	±2.726	46	0.9306	33.738	±2.051
47	0.3840	19.229	±2.687	47	0.9347	33.885	±2.407
48	0.3750	18.781	±2.834	48	0.9315	33.768	±2.481
49	0.3761	18.829	±2.672	49	0.9318	33.778	±2.433
50	0.3802	19.025	±2.737	50	0.9284	33.663	±2.530
51	0.3705	18.551	±2.753	51	0.9252	33.544	±2.546
52	0.3722	18.634	±2.661	52	0.9229	33.461	±2.464
53	0.3732	18.684	±2.834	53	0.9212	33.392	±2.501
54	0.3721	18.627	±2.775	54	0.9184	33.301	±2.635
55	0.3710	18.565	±2.620	55	0.9216	33.410	±2.624
56	0.3775	18.891	±2.633	56	0.9192	33.323	±2.600

APPENDIX D

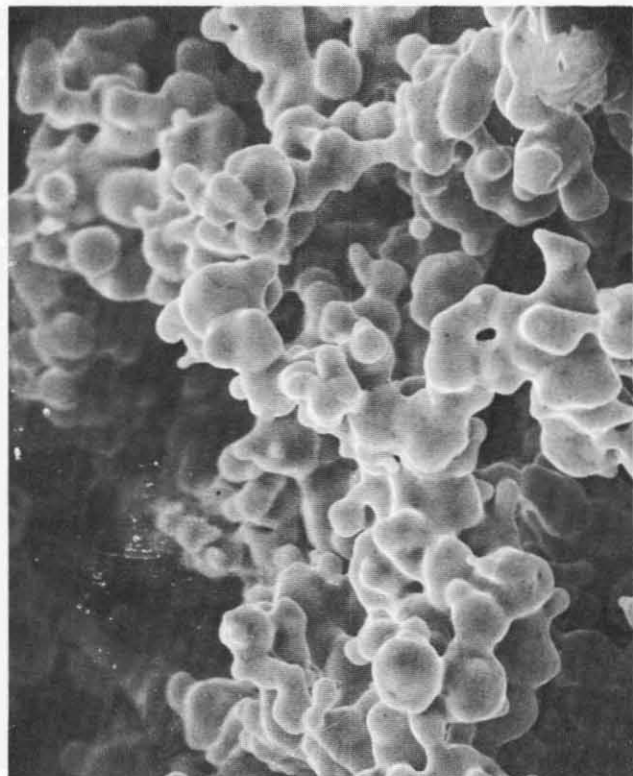
**ADDITIONAL SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUES (80 % POROSITY 15 – 90 μ)
AND ELECTRODES MADE FROM SAME**



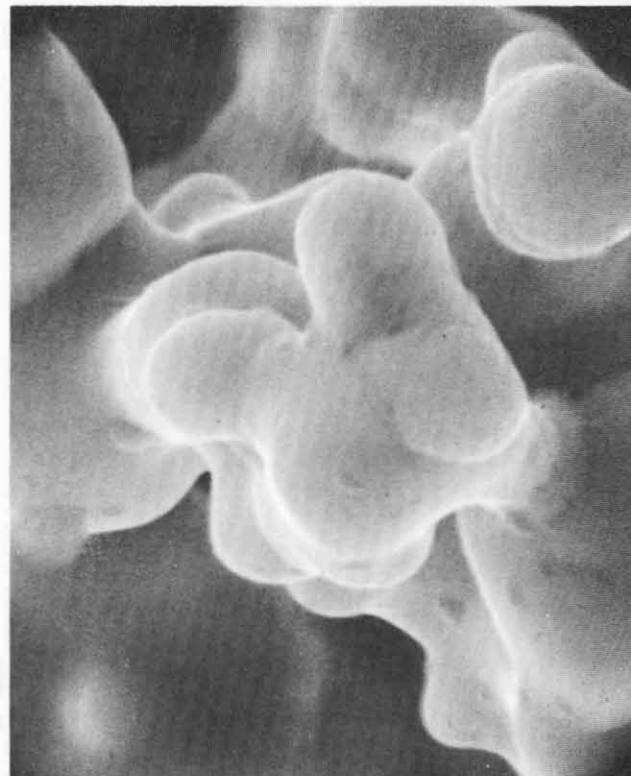
110X



1100X

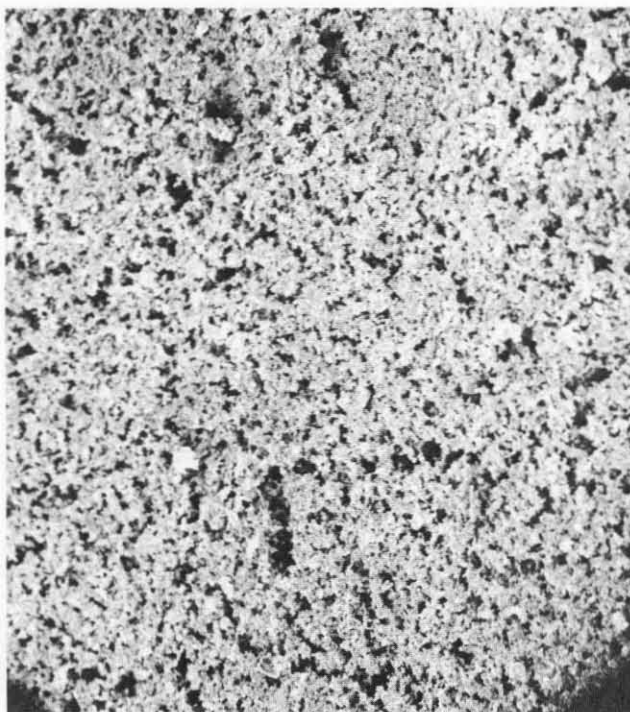


2200X

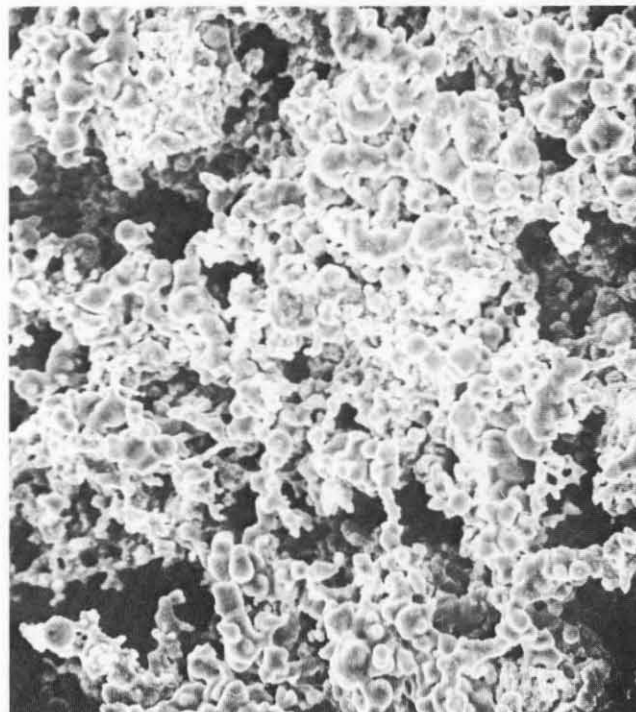


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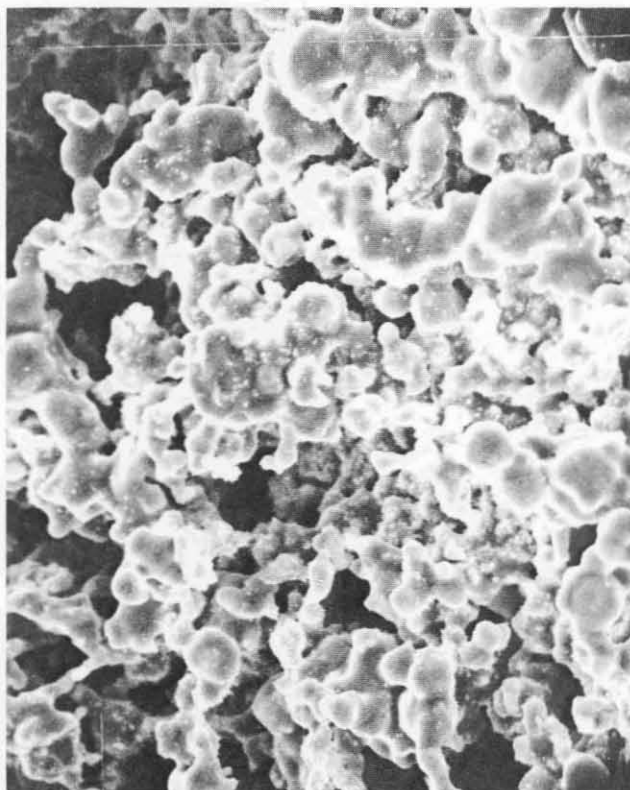
**FIGURE 33. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUES, INCO 287 POWDER 80% 15 μ**



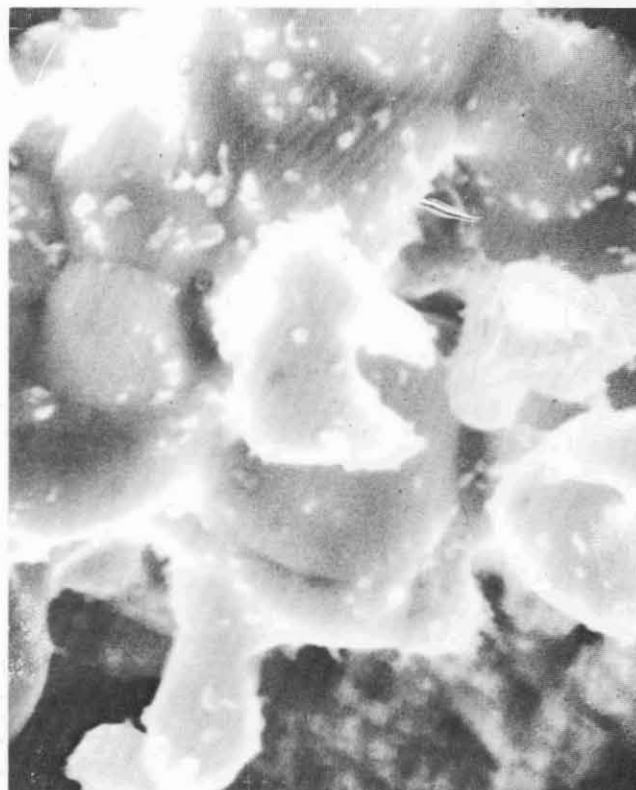
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1000X



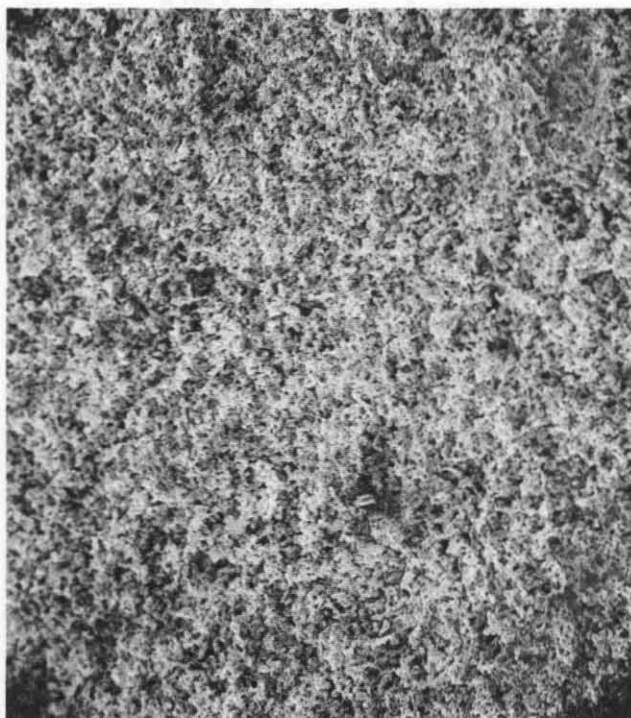
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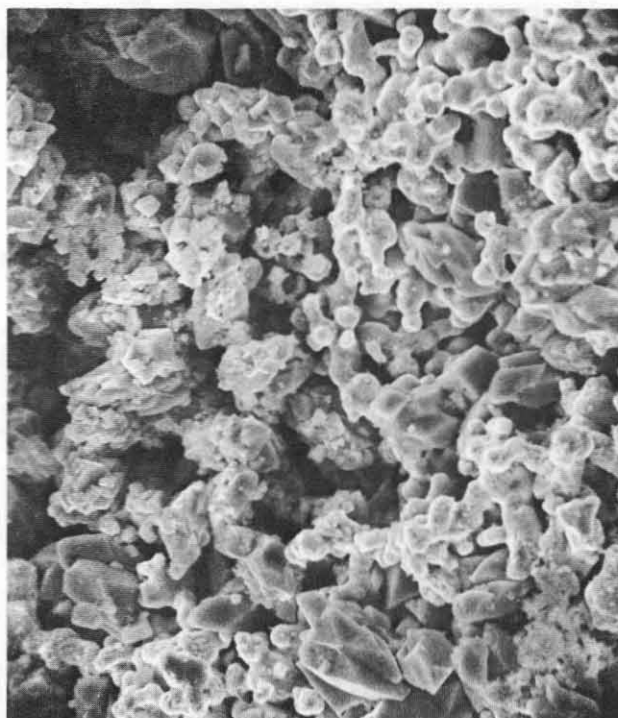
10,000X

FIGURE 34. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY COMPACTED
PLAQUE, BLENDED POWDER*, 80 % 25 μ

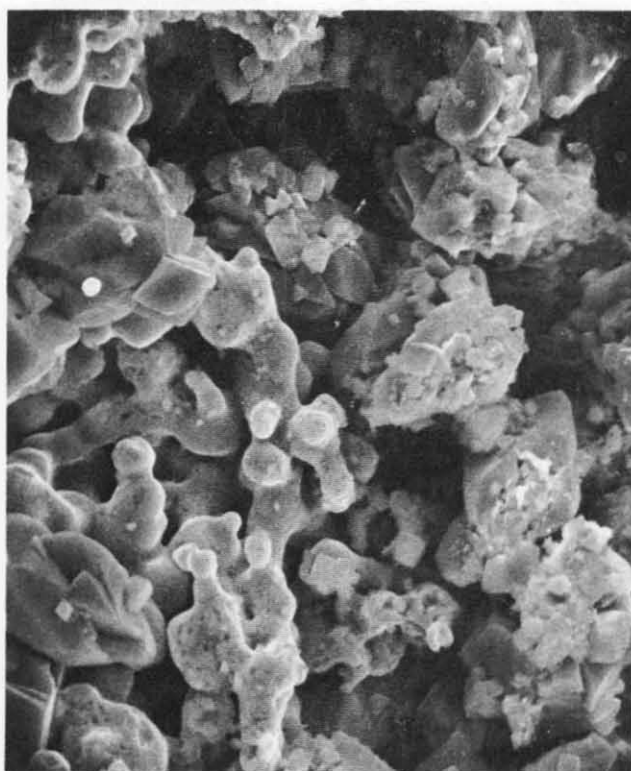
*INCO 287 and Sheritt-Gordon Powders



115X



1150X

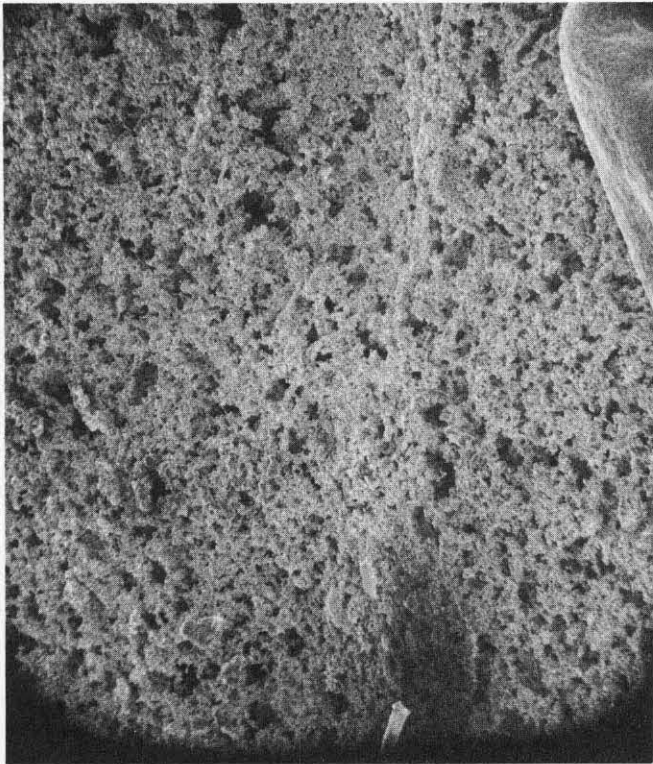


2300X

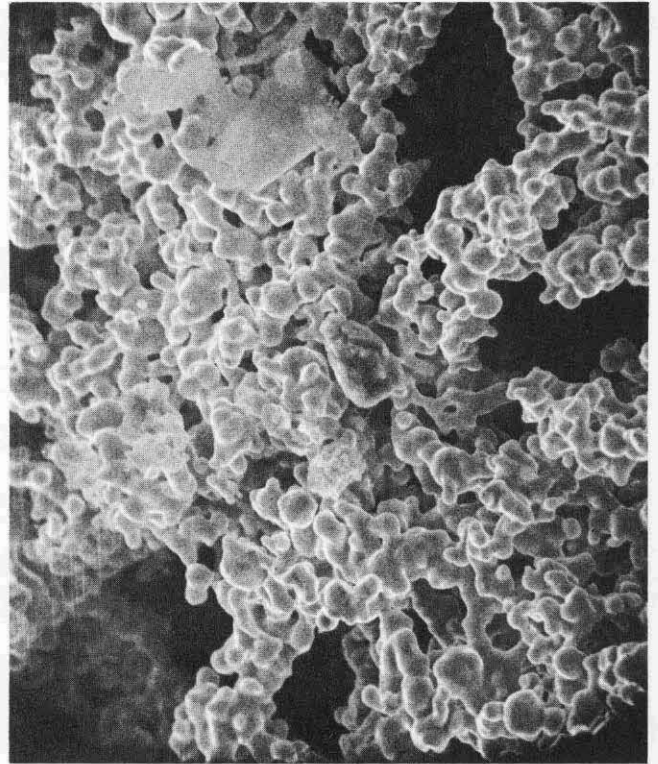


11,500X

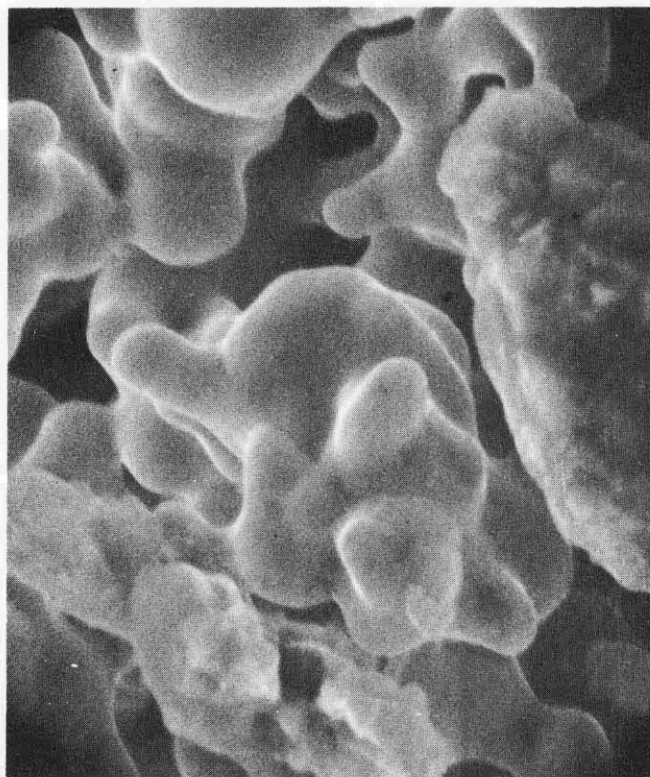
FIGURE 35. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODE MADE FROM
ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 80 % 25 μ



100X

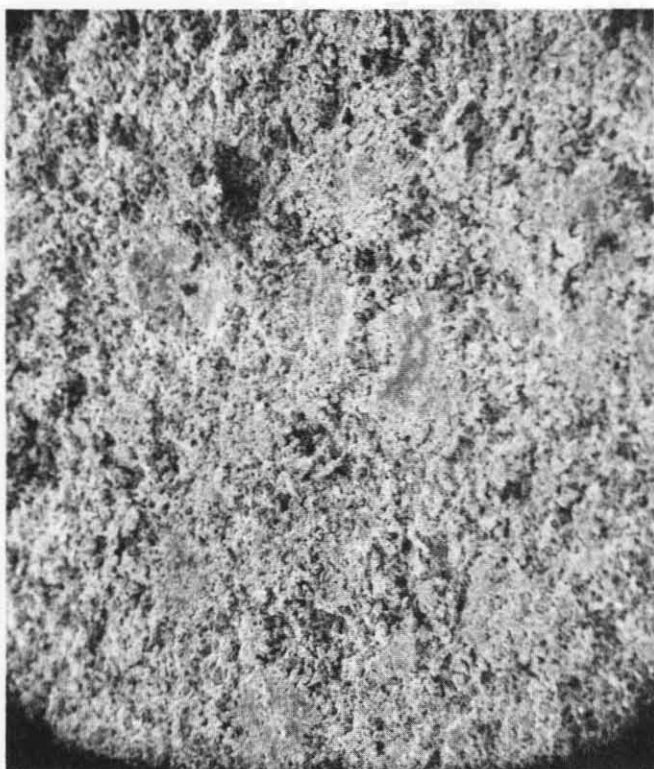


1000X

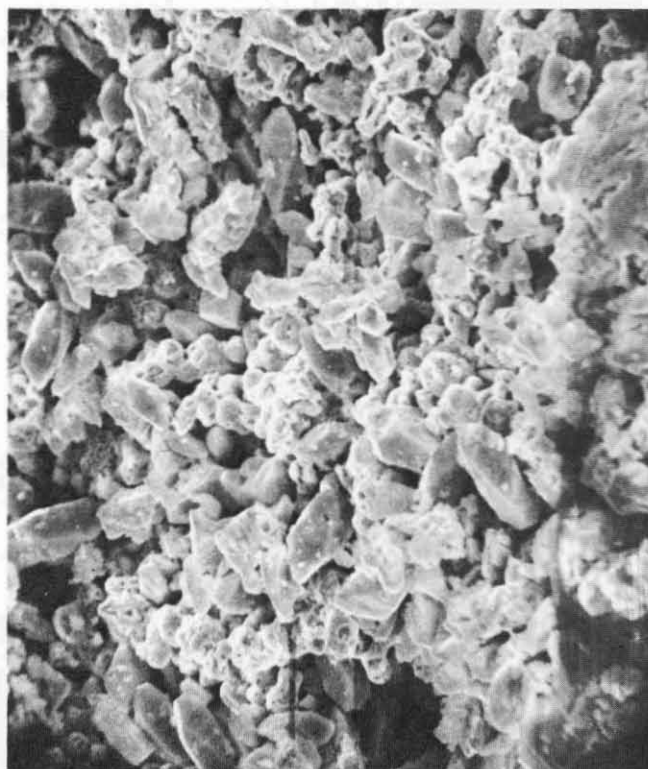


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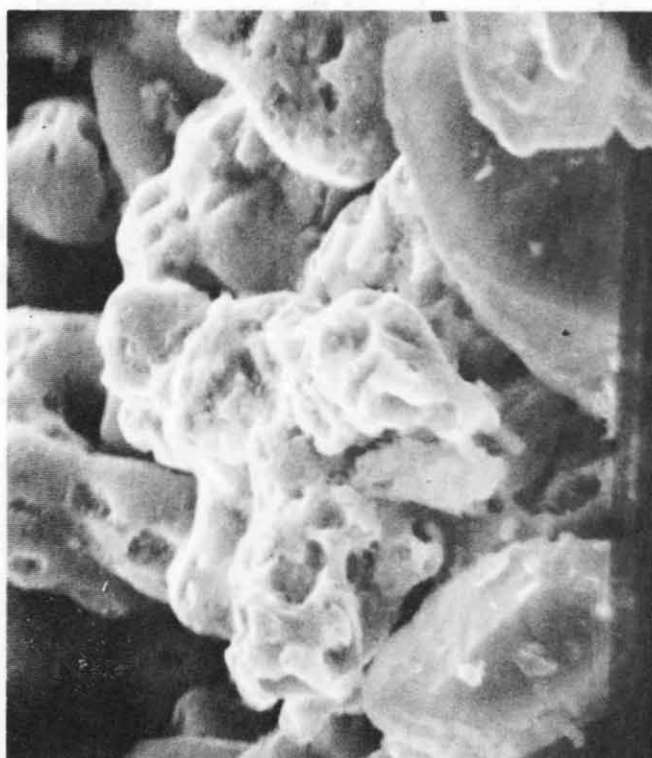
FIGURE 36. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 80 % 50 μ



101X

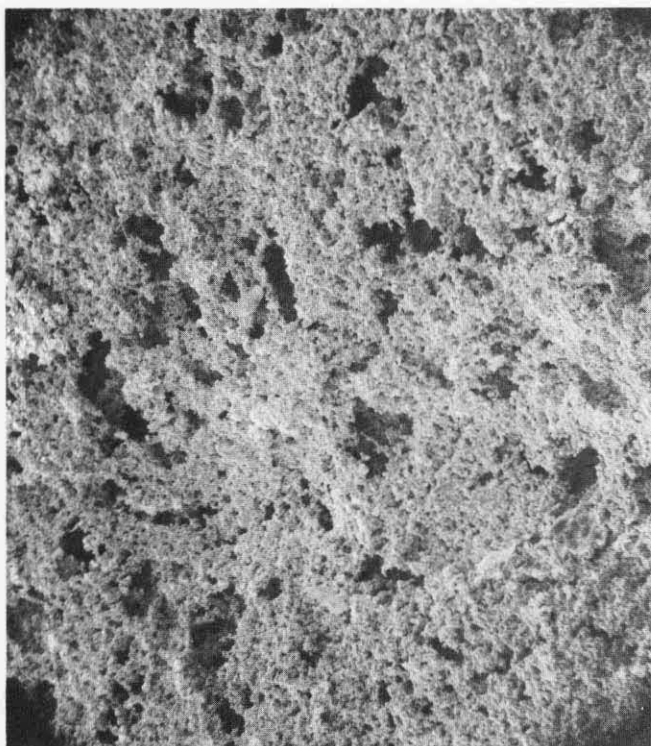


1010X

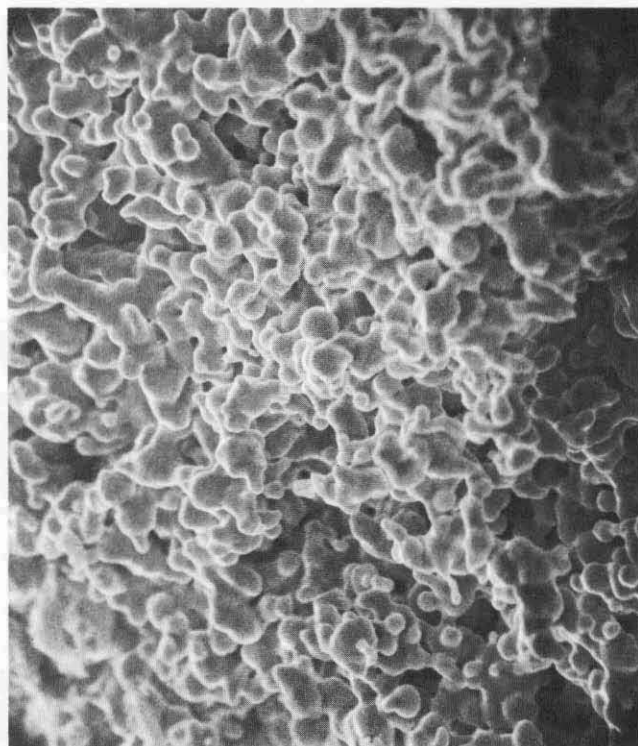


5050X

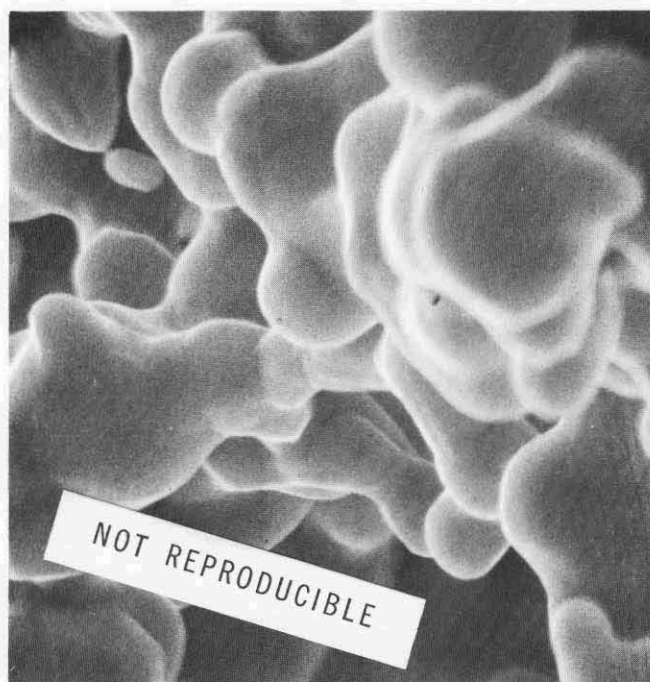
FIGURE 37. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 80 % 50 μ



125X

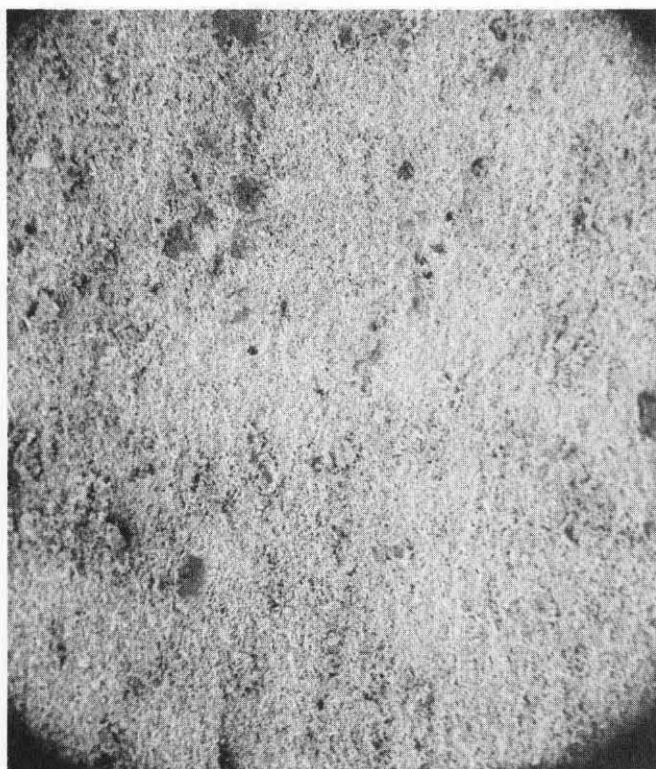


1250X

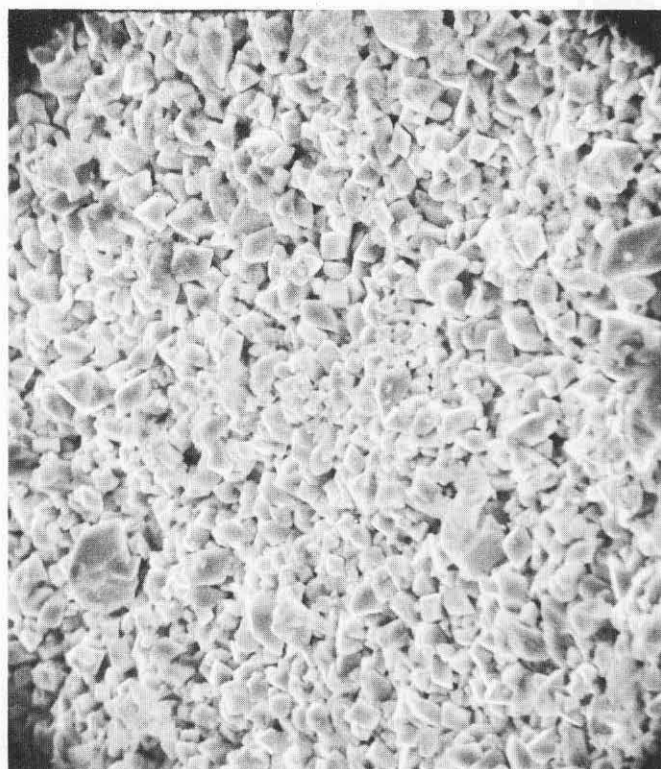


6100X

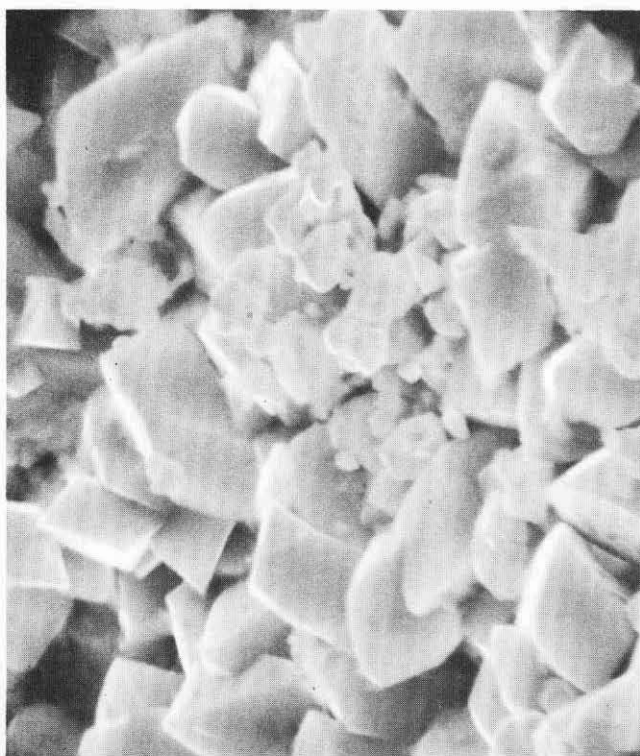
**FIGURE 38. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUE, INCO 287 POWDER, 80 % 90 μ**



92X



920X

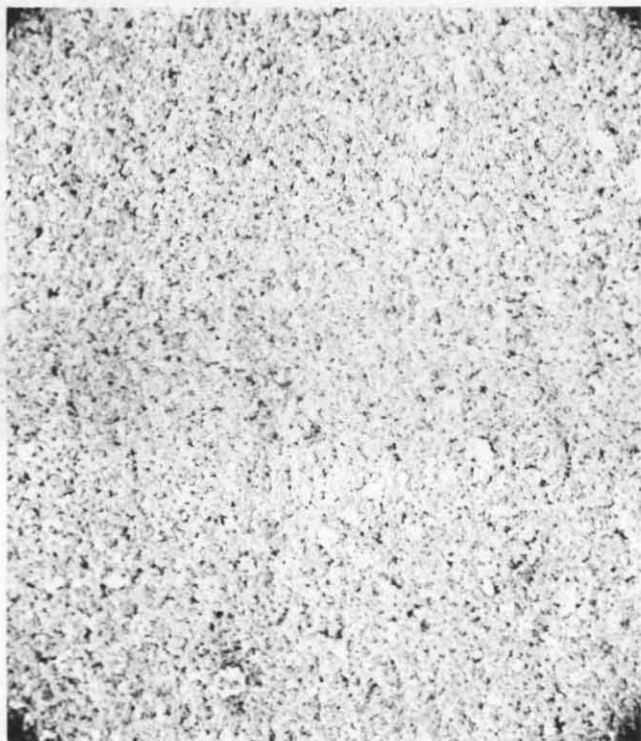


4600X

FIGURE 39. SEM PHOTOMICROGRAPHS OF FORMED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO POWDER, 80 % 90 μ

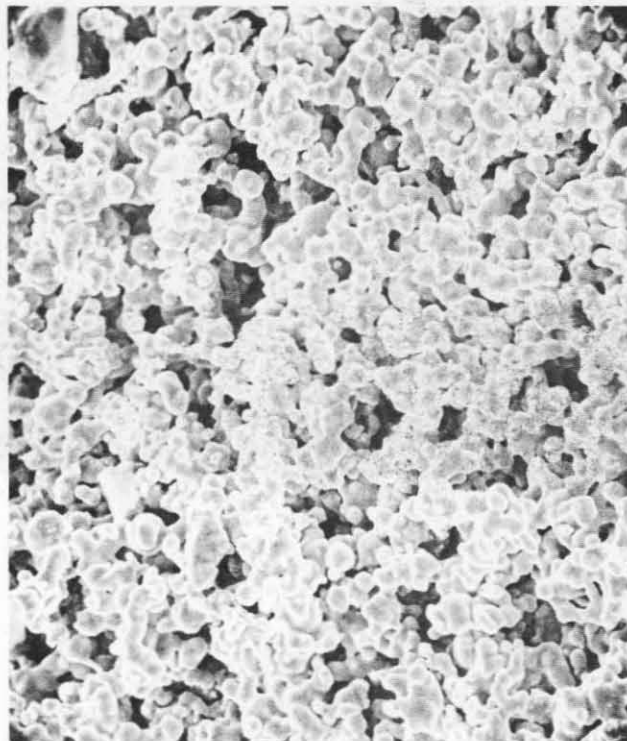
APPENDIX E

**ADDITIONAL SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUES (60 % POROSITY 15 – 90 μ)
AND ELECTRODES MADE FROM SAME**

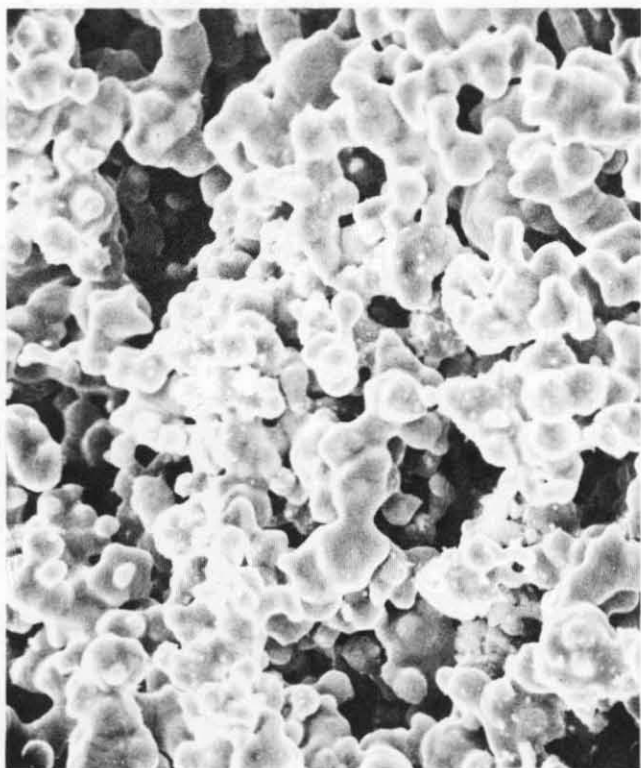


100X

Reproduced from
best available copy.



1000X

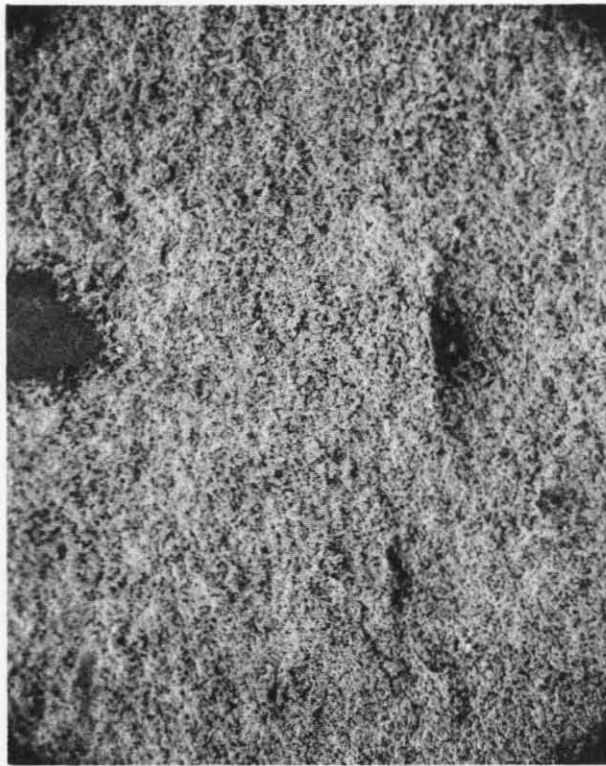


2000X

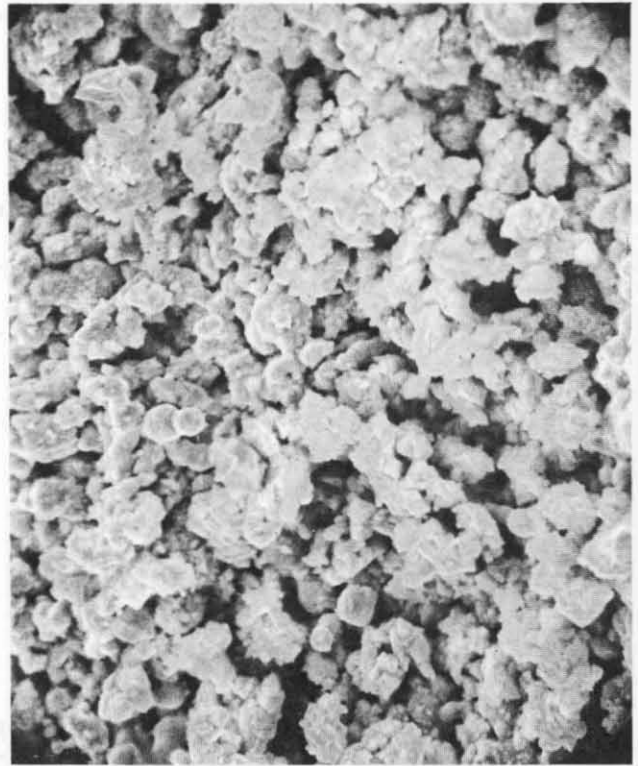


10000X

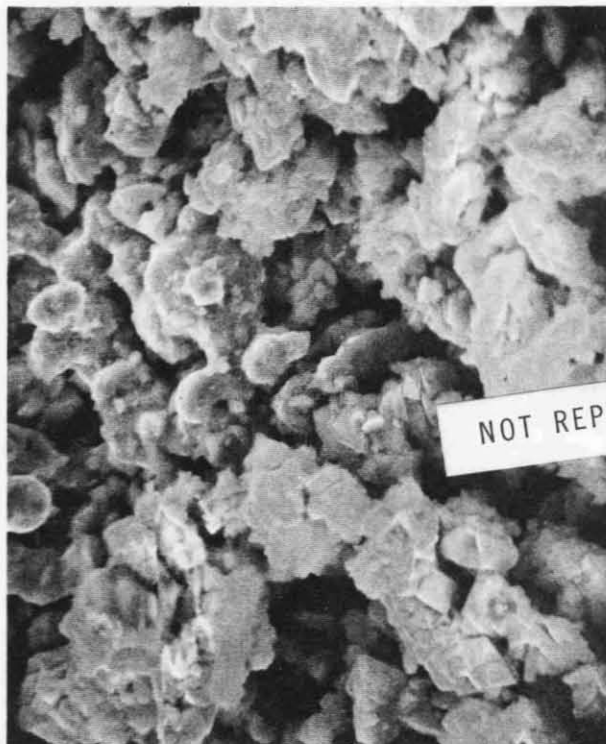
FIGURE 40. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUES, INCO 287 POWDER, 60 % 15 μ



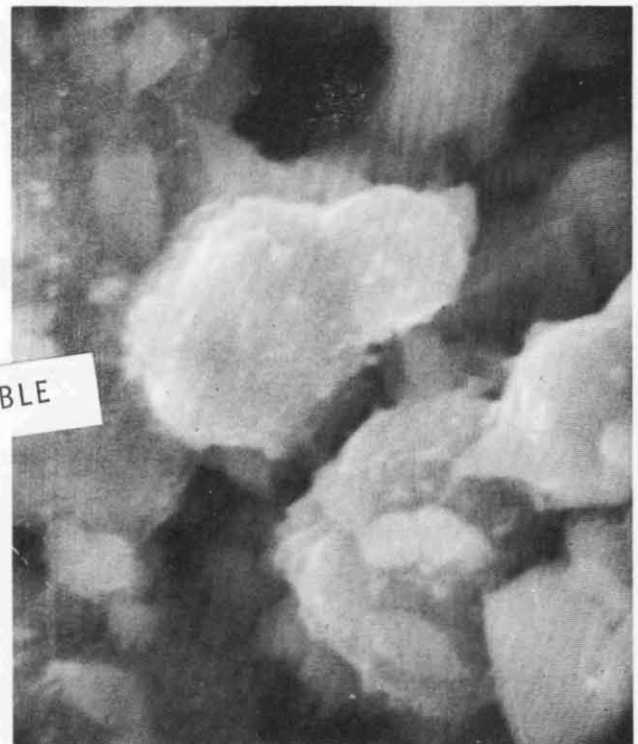
120X



1200X



2400X



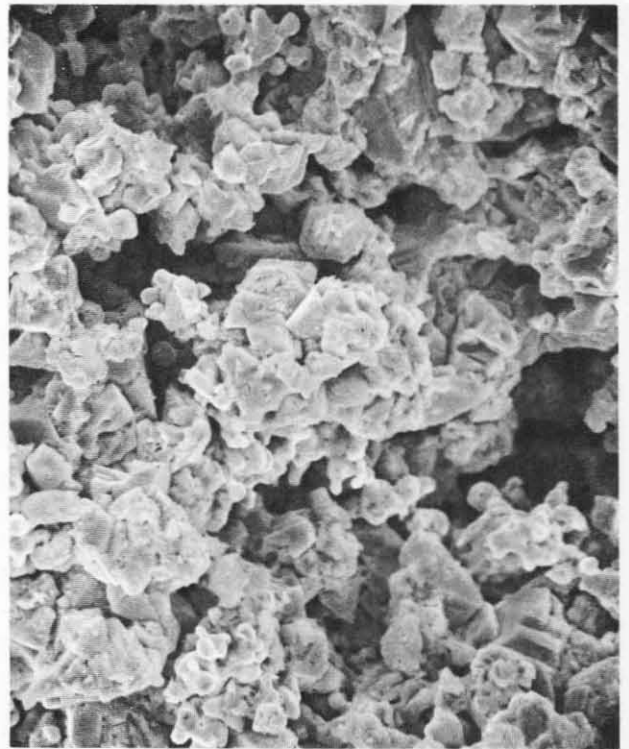
12000X

NOT REPRODUCIBLE

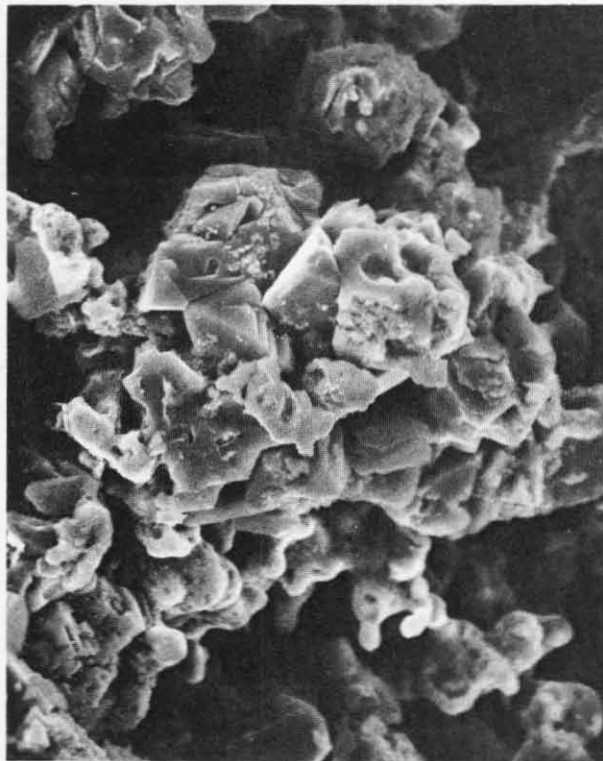
FIGURE 41. SEM MICROGRAPH OF CYCLED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 60 % 15 μ .



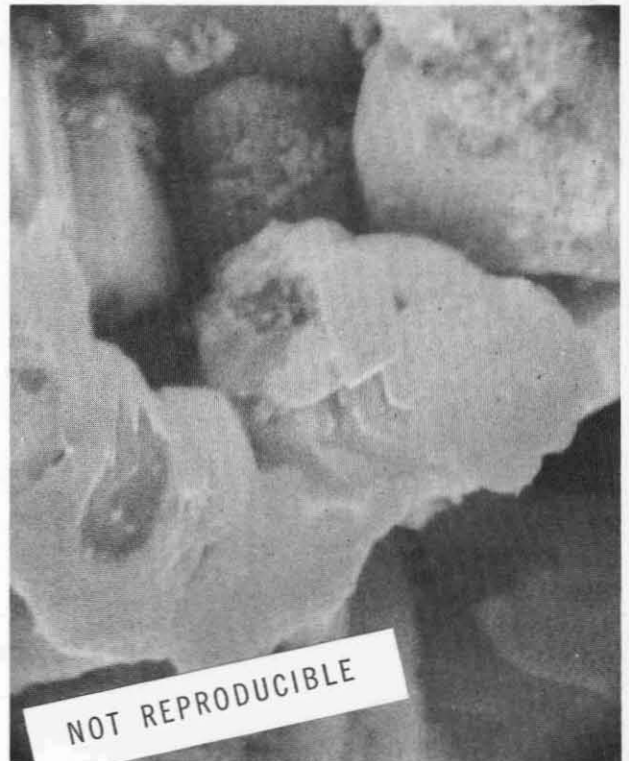
Cross Section 120X



Cross Section 1200X

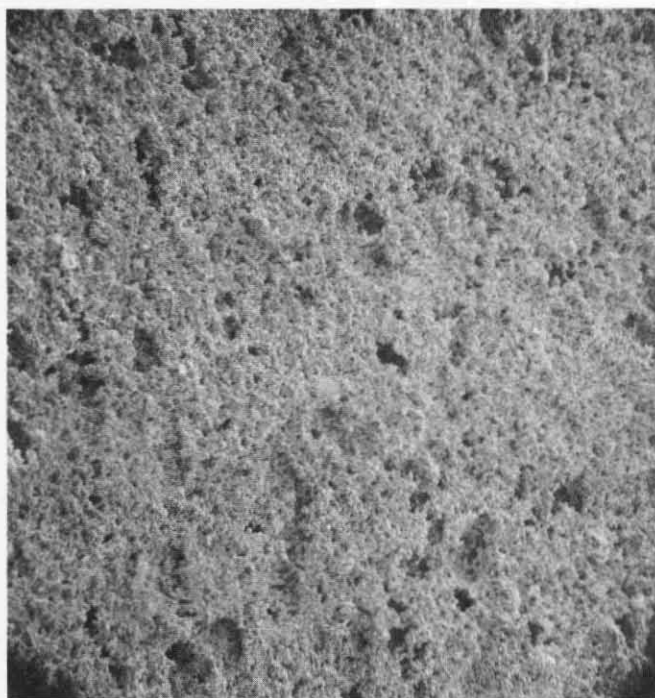


Cross Section 2400X

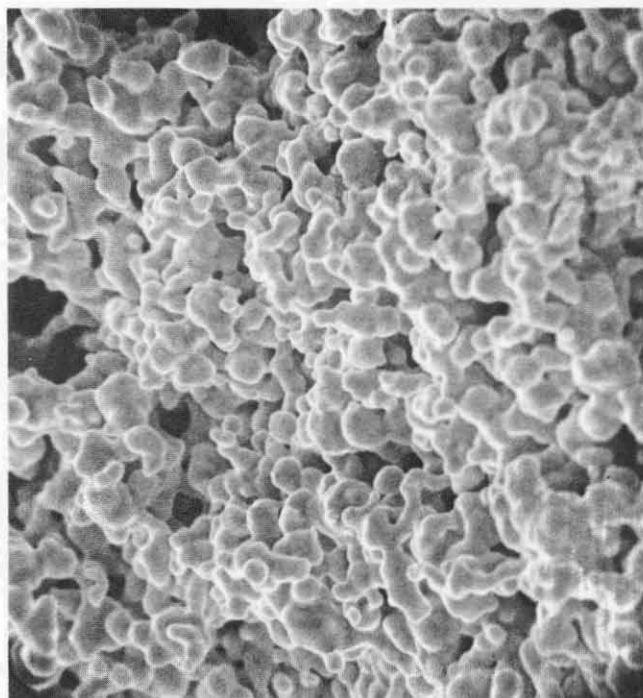


Cross Section 12000X

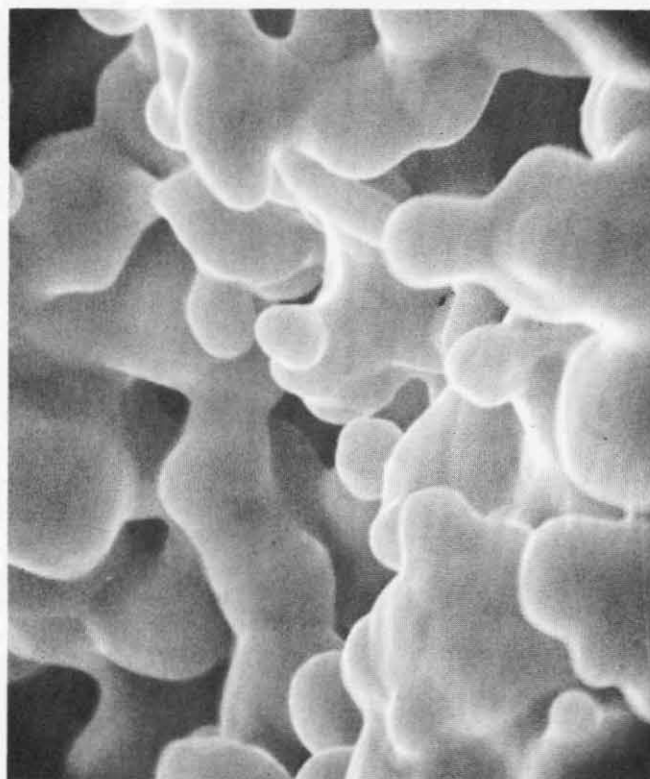
FIGURE 42. SEM PHOTOMICROGRAPHS OF CYCLE ELECTRODES MADE FROM ISOSTATICALLY COMPACTED PLAQUE, 60 % 25 μ



120X



1200X

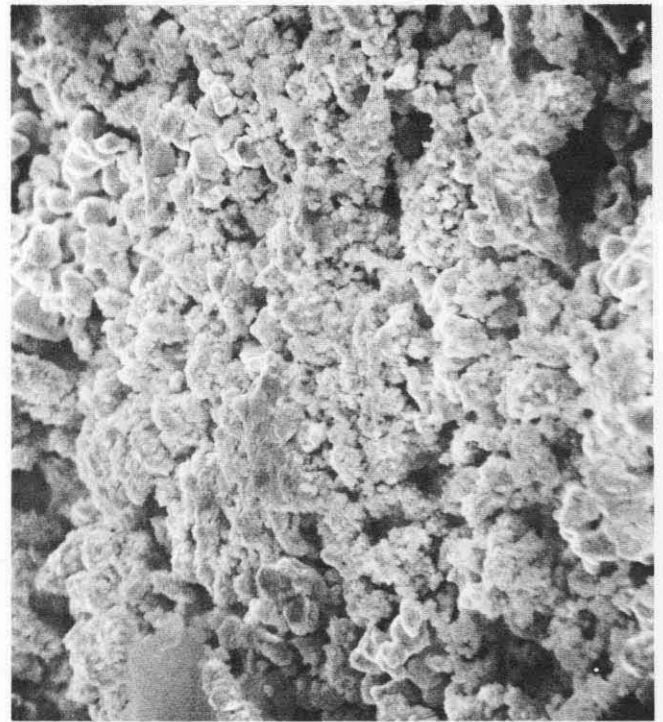


6000X

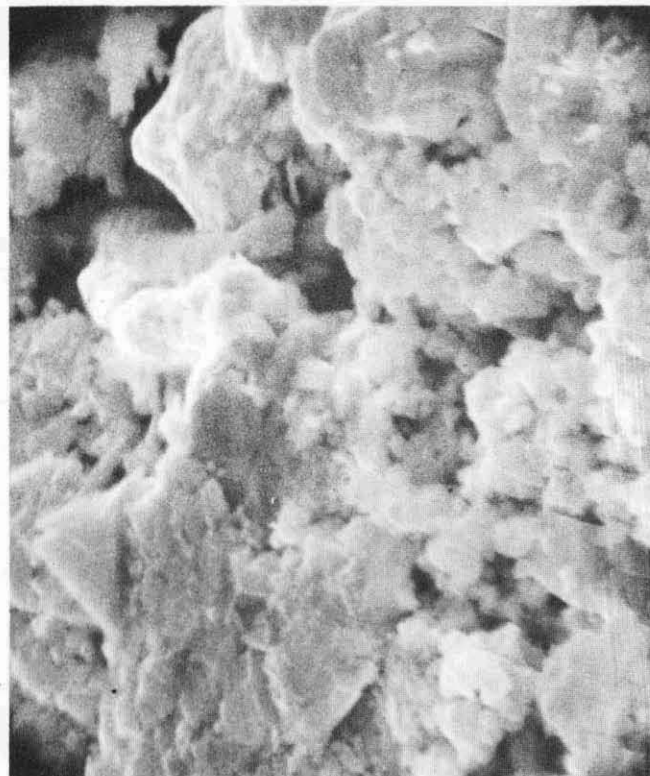
**FIGURE 43. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY COMPACTED
PLAQUE, INCO 287 POWDER, 60 % 50 μ**



100X

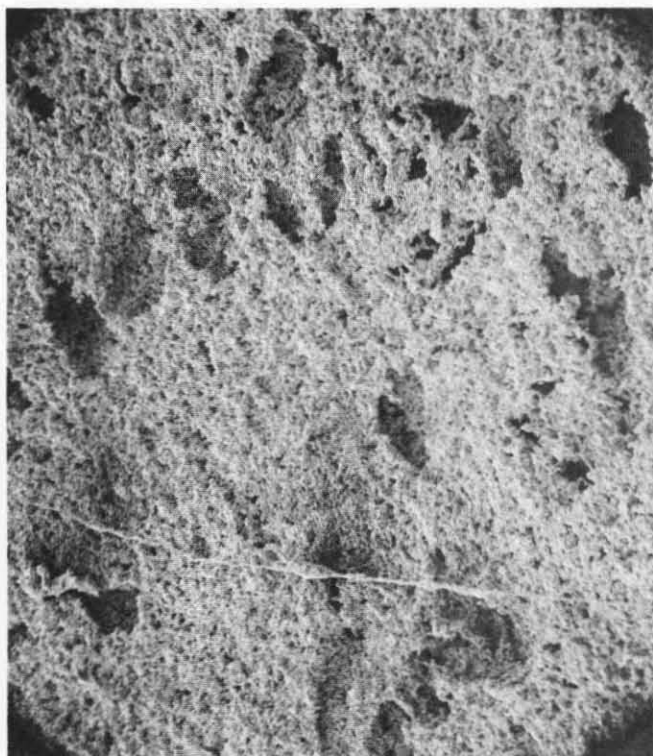


100X

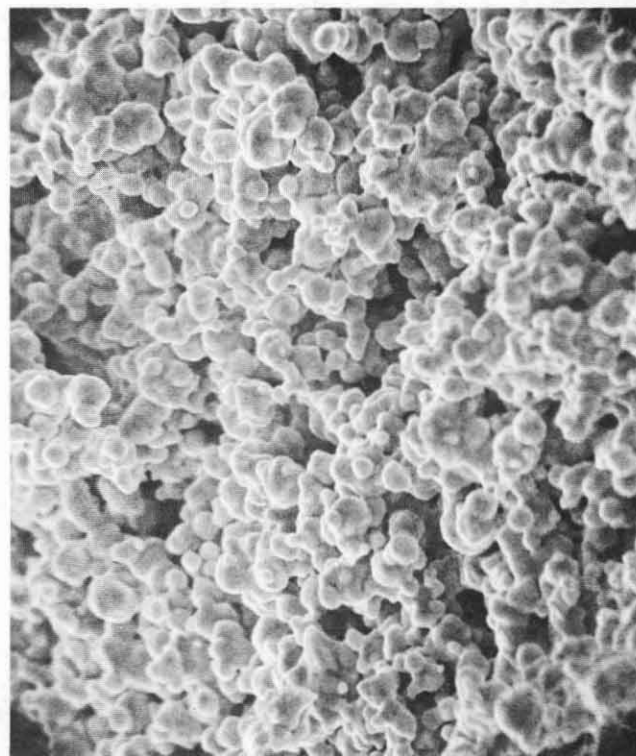


5000X

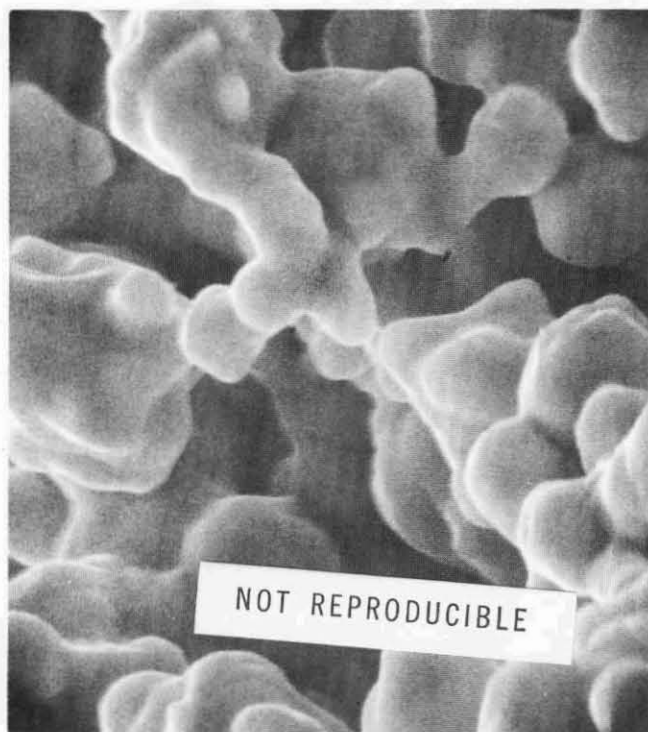
FIGURE 44. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 60 % 50 μ



126X

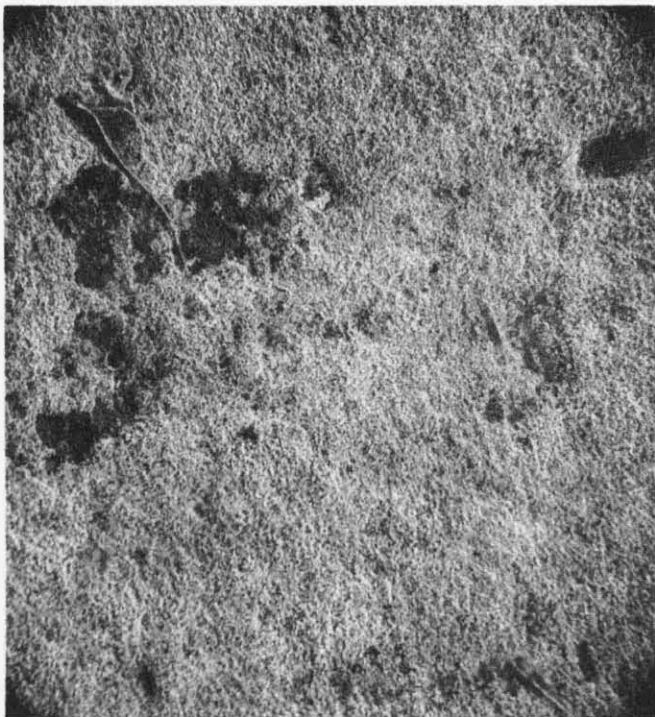


1206X

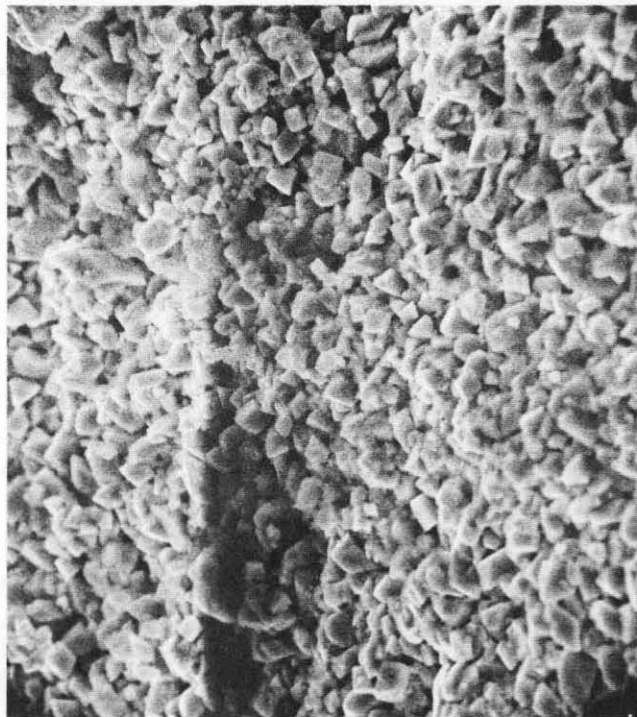


6300X

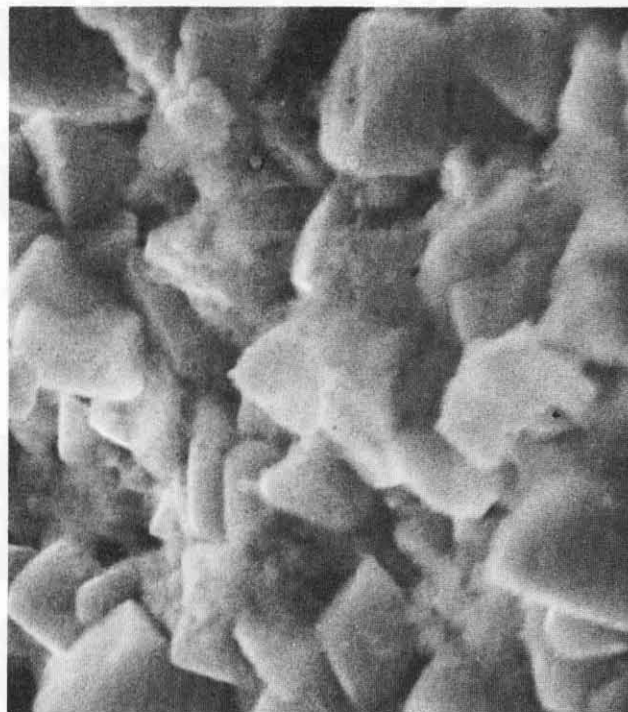
FIGURE 45. SEM PHOTOMICROGRAPHS OF ISOSTATICALLY
COMPACTED PLAQUE, INCO 287 POWDER, 60 % 90 μ



110X

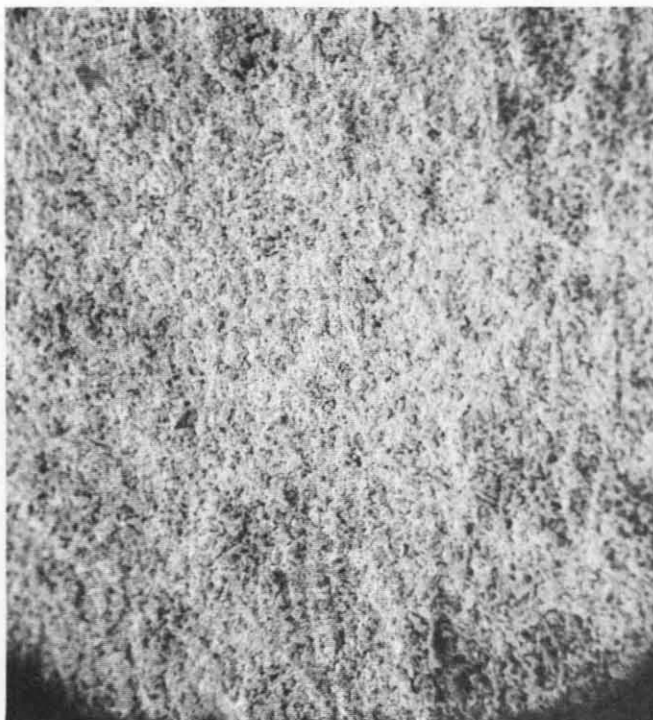


1100X

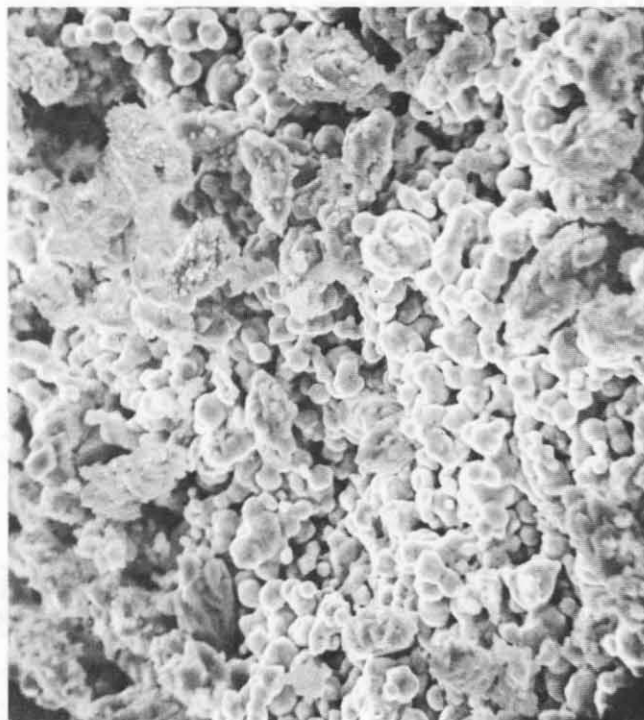


5500X

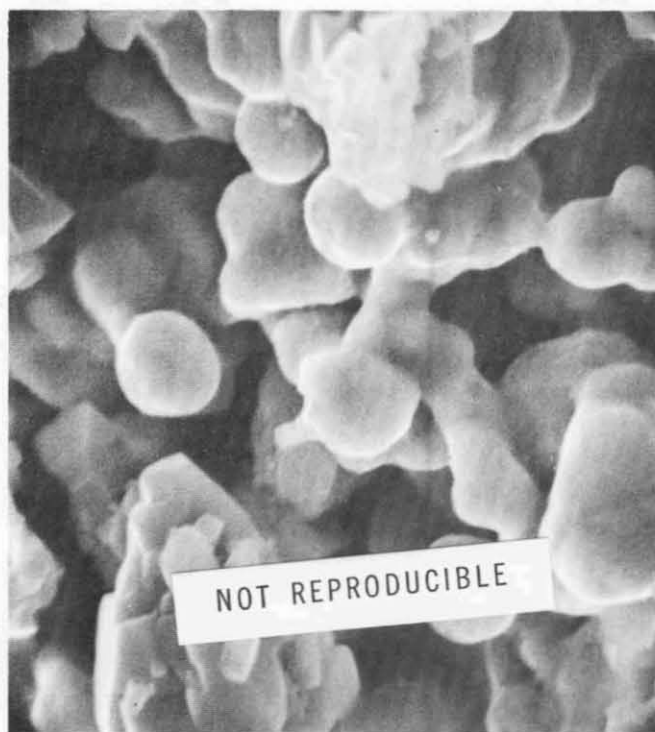
FIGURE 46. SEM PHOTOMICROGRAPHS OF FORMED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 60 % 90 μ



116X



1160X



5800X

FIGURE 47. SEM PHOTOMICROGRAPHS OF CYCLED ELECTRODE MADE FROM ISOSTATICALLY COMPACTED PLAQUE, INCO 287 POWDER, 60 % 90 μ